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MANUFACTURING METHODS FOR RODUCING L-605 HARDWARE

R. A. MASYLOW THE MARQUARDT CORPORATION

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ABSTRACT

This report summarizes a program designed to develop a process for producing large components for rocket/ramjet combustion chambers by cold shear spinning forged 1.-605 preforms. Phase I of the program involved work performed on six experimental heats of 1.-605 directed toward optimization of melting process, composition tolerances, fabricability, response to thermal treatments, and mechanical properties. This work resulted in an improved specification for 1.-605 forgings of shear spinning quality. Phase II of the program involved process development for production of SCP/LAsith combustion chamber center sections which resulted in a 1.-605 shear spinning process specification. In addition, an attempt was made to shear spin a contribugally cast preform.

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MANUFACTURING METHODS FOR PRODUCING L-605 HARDWARE

R. A. HARLOW

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FOREWORD

This Final Technical Report covers all work performed under Contract F33615-67-C-1154 from 1 October 1966 to 31 December 1967. The manuscript was released by the author in December 1967 for publication as an AFML Touhnical Report.

This contract with the Marquardt Corporation was initiated under AFML Project 873-7, "Manufacturing Methods for Producing L-605 Hard are". It was accomplished under the technical direction of Mr. John O. omyder of the Advanced Fabrication Tookergoes Deant (MATT), Monafacturing Technology Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

The program was divided into two phases:

Phase I - Preferm Variables Control

Phase II - Manufacturing Process Development

The data generated in each of these phases is presented in separate parts, but the Summary (Section II) and Conclusions (Section V) cover the entire program.

The program was directed at Marquardt by Messis. R.A. Harlow, F.K. Lampson and J.E. Widell. The following personnel were major contributors to the experimental effort:

R.D. Lloyd, J.A. Astorian and E.E. Ritchie - metallurgical analysis and evaluation; H. Gilmore - shear spinning; A.S. Rebensteine - project technician; C. A. Drury - mechanical testing; H. Binder, metallography, and J. C. Reeves, budget control.

This project has been accomplished as a part of the Air Force Manufacturing Methods Program, the primary objective of which is to develop, on a timely basis, manufacturing processes, techniques and equipment for use in economical production of USAF materials and component. .

This technical report has been reviewed and is approved.

R. MARSH, Chief

Advanced Fabrication Techniques Branch Manufacturing Technology Division

TABLE OF CONTENTS

		Page No.
SECTION		
I	INTRODUCTION	1
Ħ	Cumata	3
Ш	PHASE I - PREFORM VARIABLES CONTROL	7
	 Materials Investigated Hot Working Studies Cold Working Studies Aging Studies X-Ray Diffraction Studies Specification: "Cobalt Alloy (L-605) Forging Stock and Forgings of Shear Spinning Quality" 	8 13 23 33 40 48
IV	PHASE II - MANUFACTURING PROCESS DEVELOPMENT	55
	 Subsize Test Cylinder Fabrication SCP/LASRM Chamber Fabrication 	55 67
	 a. Preform Fabrication b. Centrifugal Casting Procurement c. Shear Spinning d. Evaluation of Shear Spun Parts 	67 80 86 102
	3. Specification: Shear Spinning Cobalt Base Alloy (L-605)	111
v	CONCLUSIONS AND RECOMMENDATIONS	115
	REFERENCES	117
	APPENDIX I - Conversion Chart for Brinnell and Rockwell C Hardness	118

DISTRIBUTION LIST

ILLUSTRATIONS (Cont)

FIGURE NO.		PAGE NO.
35	L-605 Centrifugal Casting After First Shear Spinning Pass	101
36	Photomicrographs Showing Intergranular Cracking of Shear Spun Centrifugal Casting	103
37	Pressure Test Set-Up	107

LIST OF ILLUSTRATIONS

FIGURE NO.		PAGE NO.
1	Microstructure of As Received L-605 Bar Stock	11
2	Hot Working Studies (Flow Diagram)	14
3	Examples of Forged Reductions of L-605 Bar Stock	15
4	Examples of L-605 Bars Which Cracked During Forging	17
5	Effect of Hot Working on Microstructure of L-605	19
6	Effect of Low Temperature Hot Working on the Microstructure of L-605	21
7	Cold Working Data	26
8	Effect of Cold Working and Annealing on Microstructure of L-605	31
9	Effect of Aging Treatments on the Yield Strength of 25% Cold Worked L-605 Bar	36
10	Effect of Aging Treatments on the Ultimate Tensile Strength of 25% Cold Worked L-605 Bar	36,
11	Effect of Aging Treatments on the Ductility of 25% Cold Worked L-605 Bar	37
12	Effect of Aging Treatments on the Yield Strength of 40% Cold Worked L-605 Bar	37
13	Effect of Aging Treatments on the Ultimate Tensile Strength of 40% Cold Worked L-605 Bar	38
14	Effect of Aging Treatments on the Ductility of 40% Cold Worked L-605 Bar	38
15	Effect of Cold Working and Aging on the Micro-	41

ILLUSTRATIONS (Cont)

FIGURE NO.		PAGE NO.
16	Test Cylinder Freform Forging	56
17	Microstructure of Annealed Subsize Test Cylinder Preform	59
18	Floturn Machine	63
19	Shear Spun Test Cylinders	65
20	I. D. Radius Crack in Shear Spun Test Cylinder	69
21	Center Section Preform Forging	72
22	Forging Process Flow Diagram	73
23	Microstructure of as-Received SCP/LASRM Preform	75
24	Microstructure of Annealed SCP/LASRM Preform	75
25	Center Section Machined Preform (Sketch)	77
26	SCP/LASRM Machined, Annealed, and Grit Blasted Preform	79
27	L-605 Centrifugal Casting	81
28	Microstructure of L-605 Centrifugal Casting	85
29	Microstructure of L-605 Centrifugal Casting After Heat Treating	87
30	Shear Form Roller	90
31	Shear Form Mandrel	91
32	Shear Spinning Process Flow Diagram	92
33	SCP/LASRM As-Spun Combustion Chamber Center Section	95
34	SCP/LASRM Machined Center Section	99

LIST OF TABLES

TABLE NO.	<u> </u>	AGE NO
I	Chemical Analysis of L-605 Special Melts	4
п	Data on As-Received L-605 Special Melt Forging Stock	10
ш	Room Temperature Tensile Properties of Hot Worked L-605 Bar	24
IV	Room Temperature Ductility and Hardness of Hot Worked L-605 Bar	25
v	Room Temperature Tensile Properties of Cold Worked Annealed, and Aged L-605 Bar	, 2 8
VI	Room Temperature Ductility and Hardness of Cold Worked, Annealed, and Aged L-605 Bar	29
vn	Room Temperature Tensile Properties of Cold Worked and Aged L-605 Bar	34
VIII	Room Temperature Ductility and Hardness of Cold Worked and Aged L-605 Bar	35
IX	Notched Tensile strength of Cold Worked and Cold Worked Plus Aged L-605 Bar	39
x	X-Ray Diffraction Data	43
XI	Composition of L-605 Forging Stock	51
XII	Tensile Properties of Shear Spun Test Cylinders	71
хш	Composition of Centrifugal Casting	83
хIV	SCP/LASRM Shear Spinning Data	93
xv	Dimensions of Shear Spun Parts	98
XVI	Tensile Properties SCP/LASRM Chamber	105

SECTION I

INTRODUCTION

L-605, a cobalt base superalloy, has been widely used in industry for some time, and in recent years its high strength and oxidation resistance have made it attractive for numerous prospace applications such as combustion chambers and tail pipes. At Marquardt, L-605 has been used very successfully in the cold worked condition for SCP/LASRM combustion chamber components. These components were designed to utilize the high strength to weight ratios that can be induced in this material by cold shear spinning. However, attempts to produce these parts by cold shear spinning forged preforms met with considerable difficulty due to gross cracking either during shear spinning or during a final expanding operation. Initially three parts were spun with no difficulty but subsequently, only 5 out of 20 attempts were successful. Attempts to resolve this problem by the material supplier, Marquardt, and AFML personnel were unsuccessful, which resulted in deferring this technique until additional technology could be established.

In October 1966, Marquardt, under contract to the Air Force, initiated a two phase program to extend state-of-the-art processing parameters for L-605 particularly as related to cold shear spinning. Phase I consisted of an investigation to optimize alloy composition, melting process, hot and cold working parameters, and thermal treatments for purposes of generating improved materials and process specifications. Phase II involved production and evaluation of SCP/LASRM combustion chamber center sections by cold shear spinning forged preforms according to the information developed in Phase I.

SECTION II

SUMMARY

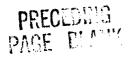
The objectives of the program were.

- Evaluate effects of melting process, composition tolerances, hot and cold working parameters, and thermal treatments on the fabricability and mechanical properties of L-605.
- Develop a manufacturing process for producing cold shear spun U.-606 hardware and generate related material and process specifications.

In view of the unexplained difficulties encountered in cold shear spinning 15 out of 20 SCP/LASRM combustion chamber center sections from forged preforms, the initial objective of the program was directed toward developing a better understanding of the metallurgy of L-605. This was considered necessary in order to optimize the quality of the forced preform, and to recommend improved processing parameters for forging and shear spinning. The second objective involved development and demonstration of an acceptable process for producing the SCP/LASRM components, and to generate specifications useful as a guide for producing these and similar parts from L-605 forgings.

To accomplish the initial objective, five special heats of varying iron, silicon, and manganese contents, (described in Table I) were supplied by the Stellite Division of Union Carbide in the form of 3/4 in. x 1 1/4 in. forged bar. These heats were vacuum induction plus vacuum arc remeited. A sixth heat was supplied in the form of 1 in. x 1 1/4 in. bar from an air melted plus vacuum arc remeited production heat. Hot working, cold working, aging, and x-ray diffraction studies were performed on these heats of material in efforts to generate data applicable to an improved specification for L-605 forgings and forging stock of shear spinning quality.





TABLE

CHEMICAL ANALYSES OF 1-605 SPECIAL MELTS

Elen	Elements (w/o)			Heat Bumbers	*		
		-41	71	mI	ঝা	ΝI	nol
O	Requested Received	.10 max.	.10 mex.	.10 max.	.10 mex.	.10 mx. .06, .054	. 156 168 168
S	Requested Received	.25 ± .10	.25 ± .30	1.0 ± 10 $91, 92$.04 max.	.25 ± .30	. S
F.	Requested Received	3.0, 1.2	$1.0 \pm .2$ $1.15, 1.38$	$1.0 \pm .2$ $1.28, \pm .15$	$3.0 \pm .2$ $2.88, \pm .23$	1.01 1.01 1.01 1.03 1.03 1.03 1.03 1.03	3.0 ± .2 2.80
<u>ru</u>	Requested Received	.01 max.	.01 max.	.01 max.	.01 max.	.01 max. .005, .005	10. 816.
S	Requested Received	.015 max.	.015 max.	.015 mex. 708, .010	.015 max.	.015 max. .012, .011	.015 max.
Mn	Requested Received	$1.40 \pm .10$ $1.32, 1.40$	$1.40 \pm .10$ $1.56, 1.22$	$1.40 \pm .10$ $1.12, \pm .16$	1.40 ± .10 1.14, 1.30	2.8 3.1.1.8 3.1.1.8	27 - 187 - 1
Cr	Requested Received	20.0 ± 1.0 $20.49, 20.45$	20.0 ± 1.0 $20.36, 20.22$	20.0 ± 1.0 $20.29, 20.29$	20.02 ± 1.0 20.02, 20.02	26.0 ± 1.0 26.22, 26.02	27 17 18 18 18 18 18 18 18 18 18 18 18 18 18
Ni	Requested Received	10.0 ± 1.0 $10.36, 10.60$	10.0 ± 1.0 $10.12, \pm 10.00$	10.0 ± 1.0 $10.20, 10.04$	10.0 ± 1.0 10.08, 10.08	10.9 ± 1.0 10.90, 9.92	100
3	Requested Received	$\begin{array}{c} 15.0 \pm 1.0 \\ 15.13 \end{array}$	15.0 ± 1.0 $14.81, 15.12$	15.0 ± 1.0 $13.98, 15.14$	15.0 ± 1.0 14.54, 15.25	15.0 ± 1.0 15.04, 15.00	11 (N)
S	Requested Received	Balance Balance	Balance Balance	Balance Balance	Balance Balance	3.2.1.2.0.ce 3.6.1.2.0.ce	हें हैं इस्ते हैं इस्ते हैं

^{*1-5} Vacuum Induction + VAR.

⁶ Air Melt + VAR.

• Hot Working Studies

Barstock from each heat was not forged into paneskes using 25% and 50% multiple reductions at temperatures of 1700, 1850, 2000, and 2150°F. The microstructure, hardness, and room temperature tensile properties of the paneske samples were evaluated and compared. All heats appeared equivalent with respect to hot workshility except the high silicon (~0.9 w/o Si) heat and the air melted plus vacuum are remelted heat, which caused some difficulty at the lower temperatures, 1700 and 1850°F. The optimum forging temperature to minimize grain boundary carbides and control grain growth appeared to be about 2150°F.

• Cold Working Studies

Cold working studies were performed by cold rolling specimens from each heat using 25 and 40% multiple reductions with interstage annealing treatments of one hour at 2000, 2150, and 2250°F. The high silicon heat was significantly the most difficult to fabricate. An evaluation of microstructure, hardness, and room temperature tensile properties was performed on material from each processing variable. It was shown that an annealing temperature in the 2150-2200°F range followed by a water quench, appeared optimum for simultaneously minimizing hardness, grain growth, and grain boundary precipitates.

Aging Studies

Samples from each heat were aged after single and multiple cold reductions of 25 and 40%. Aging treatments of 700, 1200, 1350, 1650, and 1800°F for periods of 1, 10, and 100 hours were performed. Tensile and hardness tests were run on material from each variable, and notched tensile tests were performed on selected samples. An aging treatment of 700°F for 10 hours appeared optimum for a maximum strength increase at a minimum sacrifice in ductility or notch sensitivity.

X-Ray Diffraction Studies

Precipitation of a Laves phase and complex carbides (M₂₃ C₆ and M₆C) during aging have been shown to be a contributing factor in the loss of ductility in L-605. Selected samples representing various hot and cold working conditions with subsequent annealing and aging treatments were evaluated by electrochemical extraction of the precipitates and identification by x-ray diffraction techniques. Results of the hot working and annealing studies indicate that 2150°F is sufficient to maintain the embrittling precipitates in solution. The aging treatment of 10 hours at 700°F recommended for the shear spun cylinders showed a randomly dispersed Laves phase which could not be detected using standard metallography techniques.

The second objective of the program was accomplished by fabrication of L-605 components by cold shear spinning forged preforms, utilizing the technology generated from the work involving the six experimental heats.

Initially five small test cylinders were cold shear spun from a cup shaped die forged preform, utilizing a single 50% reduction. Evaluation of these cylinders was performed utilizing dye penetrant inspection, hardness testing, and tensile tests. The results were used primarily to verify the shear spinning quality of the forged preform. Upon successful completion of this effort, five large cup shaped die forgings (SCP/LASRM chamber preforms) were procured from the same heat of material and to the same specification. The preforms were successfully shear spun to approximately 15 in. diameter x 32 in. long x 0.125 in. wall cylinders, using a four step shear spinning process. The quality of the parts was verified by dye penetrant and ultrasonic inspection, hydrostatic pressure proof tests, and tensile tests.

The results of the experimental heat investigation and shear spinning process development were summarized in useable form within two specifications included in this report:

- (1) Cobalt Alloy (L-605) Forging Stock and Forgings of Shear Spinning Quality.
- (2) Shear Spinning of Cobalt Base Alloy (L-605).

SECTION III

PHASE I - PREFORM VARIABLES CONTROL

In view of the past difficulties encountered in shear spinning and expanding the SCP/LASRM combustion chamber center sections (as explained in Section I), it was believed necessary to develop an improved forged preform of consistent shear spinning quality as the initial step in solving the problem. A failure analysis performed by the Materials Application Division of the Air Force Materials Laboratory on cracked cases resulted in data regarding the nature of the failure, which was attributed to an overload condition. However, this information did not explain why some parts failed and some were produced without difficulty. The 15 failures out of 20 attempts could not be correlated with a specific heat of material or processing variable by Marquardt or others involved in the problem.

In order to develop a specification for a forged preform of shear spinning quality, the melting process was considered to be of importance, i.e., air melt plus vacuum arc remelt vs. vacuum induction plus vacuum arc remelt. These two processes are presently used for producing L-605 commercially, and whether any significant differences with respect to cold shear spinning characteristics existed was unknown. The effects of the composition tolerances on fabricability and response to thermal treatments were also considered important in optimization of preforms. Wlodek's work (2) indicated that heats of commercial L-605 that are high in iron (2-3%) and low in silicon (0.5% or less) and carbon (less than 0.1%) appear to be less susceptable to an embrittling effect (precipitation of a Laves phase after prolonged exposure to elevated temperature). The effect of silicon content was substantiated by Sandrock (3) and Nejedlik (4), but little apparent effect of iron was noted. Although these investigations were concerned with aging treatments for extended periods of time, it was believed that composition could also affect shear spinning characteristics, particularly after multiple passes and interstage annealing treatments.

The effect of processing variables, i.e., annealing parameters, hot working variables, etc. were also considered worthy of investigation for control and repreducibility of forged preform quality. The AMS specification (No. 5759D) for L-605 forgings defines a solution heat treatment of 2250°F followed by a water quench or rapid air cool. However, subsequent work performed at Marquardt on other programs showed that a preform grain size of ASTM 3 or finer was desirable for cold shear spinning, and that a 2250°F anneal after forging caused extreme grain growth. It was found that ~2150°F annealing temperature sufficiently lowered the as forged hardness without causing excessive grain growth. Schultz (5) showed that a 2150°F anneal (1) prevented excessive grain growth, (2) stabilized microconstituents, and (3) reduced structural inhomogeneity. Because of these inconsistencies in data compared to the AMS specification, it was considered necessary to optimize post forging annealing and interstage annealing treatments for the shear spinning operations. Additional unknowns existed regarding effects of hot forging variables on subsequent cold fabricability. Also, since a target of 175 KSI yield strength existed for SCP/ LASRM, data regarding possible recommendation of an optimum post shear spinning aging treatment were desirable.

Based on the unknowns outlined above, the following program was conducted as Phase I of the contract.

1. MATERIALS INVESTIGATED

In order to further investigate the effects of melting process and composition variables, the six heats described in Table I were procured. The compositions were selected to be within the AMS 5759D tolerances, but to vary elements that had been shown to have significant effects on properties as discussed above. Heats 1 and 2 varied iron from $\sim 1\%$ to $\sim 3\%$ holding silicon low ($\sim .25\%$). Heat No. 3 held silicon at the maximum, leaving iron at relatively low, $\sim 1\%$. Heat Nos. 4 and 6 were identical except for melting process, both being very low in silicon and high in iron. Heat No. 5 was identical with No. 2 except for a higher manganese content. The manganese was varied primarily because of its effect on increasing fabricability

as verified verbally by Stellite personnel and because of its known beneficial effects in steels. The carbon was held constant at 0.10 max. for all heats, and the phosphorous and sulphur were held at 0.01% max. and 0.015% max. respectively as compared to 0.040 and 0.030% allowed in AMS-5759D. The major constituents, chromium, nickel, and tungsten were held constant at the middle of the allowable range in efforts to avoid masking effects of the minor elements.

Rectangular bars from five special heats and one commercial heat of L-605 (described in Table I) were supplied by the Stellite Division of Union Carbide Corporation, Kokomo, Indiana. Heats 1 through 5 were vacuum induction melted (at <5,4) into 6 in. diameter ingots at Stellits, then shipped to meir subcontractor, Viking Forge & Steel Company, Albany, Calif., for vacuum arc remelting (at 20-30 μ) to 8 in. diameter ingots, hot rolling and forging to rectangular bar, and annealing (2150° F for 1 hour, water quench). Due to a lower than anticipated yield on vacuum arc remelting and ingot conditioning, it was necessary for Viking to forge the bar stock to a smaller than requested cross section (3/4 in. x 1 1/4 in. vs. 1 in. x 1 1/4 in.) in order to meet the overall length requirement necessary to conduct the Phase I fabrication studies. The sixth heat (air melt plus vacuum arc remelt) was supplied as commercial material in the form of 1 in. x 1 1/4 in. rectangular bar. The microstructures of the as-received material are shown in Figure 1. It was noted that the grain size of Heat No. 3 (high silicon) was significantly finer than the heats (1, 2, 4, 5) which were processed identically. The microcleanliness data, grain size, Rockwell hardness, and tensile data are shown in Table II. The microcleanliness data on double vacuum melted heats were comparable; the air melt + vacuum arc remelted Heat No. 6 revealed only a slightly higher rating. The hardness of all heats were comparable with the exception of Heat No. 3 which contained high silicon (and revealed the smallest grain size). This heat also showed higher strength and lower elongation.

TABLE II

1							
	Elong. (3)	71*	62**	4 0**	64**	**4.9	61***
(6)	Utamate Strength	136, 500*	146,900**	159, 300**	136, 950**	137,850**	141, 700***
(g) PIOIA	Strength 2% Offset	64, 500*	65, 450**	80,300**	62,500**	64, 200**	68, 150***
	Hardness (Aversor)	R _B 95	R _B 96	R _C 29	R 95	R _B 97	R _B 98
Grain (2)	Size (Average)	4.3		œ	ထ က	4.5	တ
	Type D	0 0	0	0	0 0	0	0 0
	pe C	0	0	0	0	0	0
E,	t aft	0.6	-	 1	-	-	8
on Rath		0	0	0	0	0	0
nclus	Thu	0	•	0	•	•	0
I		0	0	0	0	0	0
	Thin	0.6		0	0.3	0	-
	Heat	-	83	က	4	ro	9
	Grain (2) Broine !! Yield (3)	Type A Type B Type C Type D Size Hardness 2% Offset Strength Thick Thin Thin Thick Thin Thin Thick Thin Thin Thin Thin Thin Thin Thin Thin	Inclusion Rating (1) Type B Type B Type D Size Hardness (Average) Rockwell Hardness (Average) Strength (Ithmate Strength (Ithma	Type A Type B Size Hardness .2% Offset Strength Ultimate (Strength 0.6 0 0 0 0 4.3 RB 95 64, i300* 136, 500* 1 0 0 0 0 3.7 RB 96 65, 450** 146, 900**	Type A Type B Type B Type B Type B Type B Type D Size Barcherage Hardness (Average) Strength (Average) Uttimate (Bis) Uttimate (Bis) Uttimate (Bis) 0.6 0 0.6 0 0 4.3 RB 95 64, i300* 136, 500* 1 0 0 0 0 0 3.7 RB 96 65, 450** 146, 900** 0 <	Inclusion Rating (1) Grain (2) Rockwell Rockwell Rockwell Rockwell Strength Strength Strength (1) Yi ild (3) Yi ild (3) Thum Thick Thir Thick Thir Thick (Average) (A	Type A Type B Type C Type D Size Hardness Case Strength Strength Uitimate (3)

- Notes:
 (1) Per ASTM E-45, Method D.
- (2) Per ASTM E-112, Intercept Method.
- (3) Reported by Stellite.

- * Average of two tests
- ** Average of four tests
- *** Single test

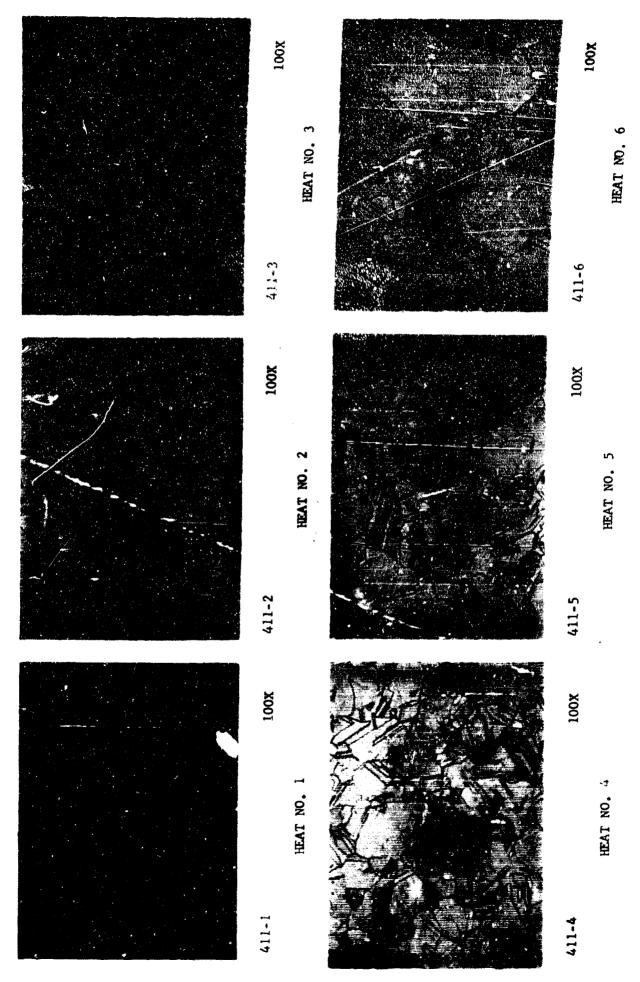


FIGURE 1. MICROSTRUCTURE OF AS-RECEIVED L-605 BAR STOCK.

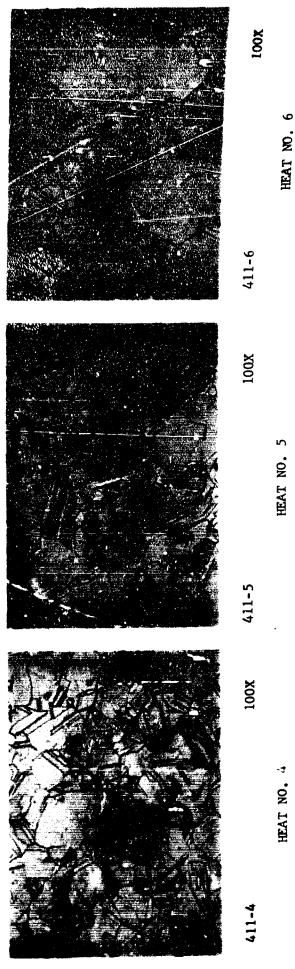


FIGURE 1. MICROSTRUCTURE OF AS-RECEIVED L-605 BAR STOCK.

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2. HOT WORKING STUDIES

In order to establish differences between the six heats with respect to hot working characteristics and to generate information regarding optimum forging temperatures, hot working studies were conducted on bar stock specimens from each of the six heats. Pancakes were forged from the bar (reducing the 1 1/4 in. dimension) using 25 and 50% multiple reductions at 1700, 1850, 2000, and 2150°F at Arcturus Manufacturing Co., Oxnard, California, as shown in the flow chart, Figure 2. Samples receiving multiple reductions at the same temperatures were returned to the furnace and reheated to temperature (measured optically) then reforged. Samples to be reforged at lower temperatures were water quenched, then reheated to the appropriate temperature. All samples were water quenched immediately after the final reduction. All heats appeared equivalent with respect to hot forgeability with the exception of heat numbers 3 and 6 which caused some difficulty at the lower temperatures, 1700 and 1850°F. Photographs of typical forged pancakes are shown in Figure 3. Figure 4 shows typical forging failures experienced on the high s licon heat (No. 3) at 1700 and 1850°F. The air melt heat (No. 6) required several reheats at these temperatures in order to accomplish the reductions without cracking. The fact that this barstock was slightly larger in cross section than that from the other heats (lin. x 1 1/4 in. vs 3/4 in. x 1 1/4 in.) may have had some effect.

Typical photomicrographs of samples receiving multiple reductions at 2150, 2000, 1850, and 1700°F are shown in Figure 5. This type of data was considered important because the large SCP/LASRM die forging cools at variable rates during forging such that portions of the part are worked at much lower temperatures than others. The typically hot worked structures showed grain refinement at the lower forging temperatures as expected. Since grain size control is considered essential for subsequent shear spinning, the lower working temperatures appear attractive; however, the lower temperatures resulted in very heavy grain boundary precipitates (Figure 6) which would be detrimental to a subsequent shear spinning operation. Although the precipitates can be taken into

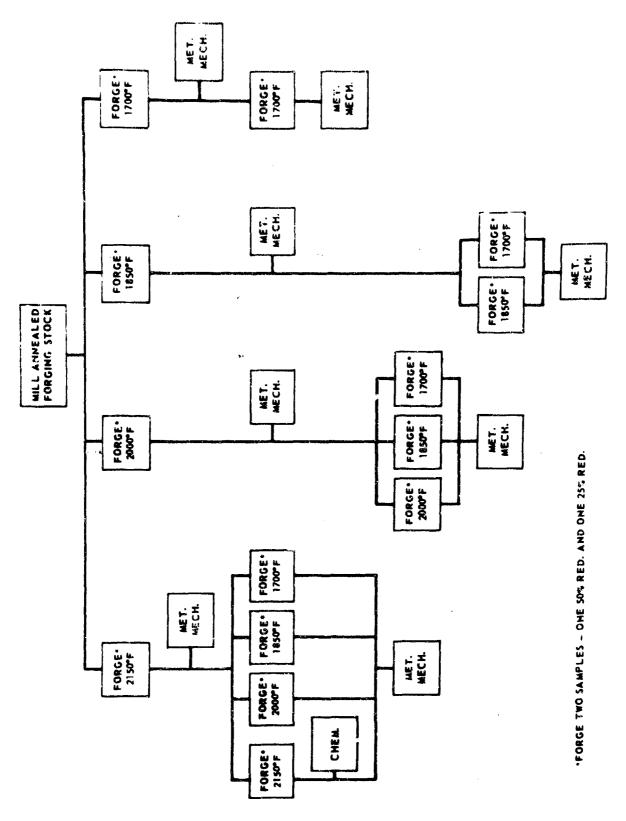
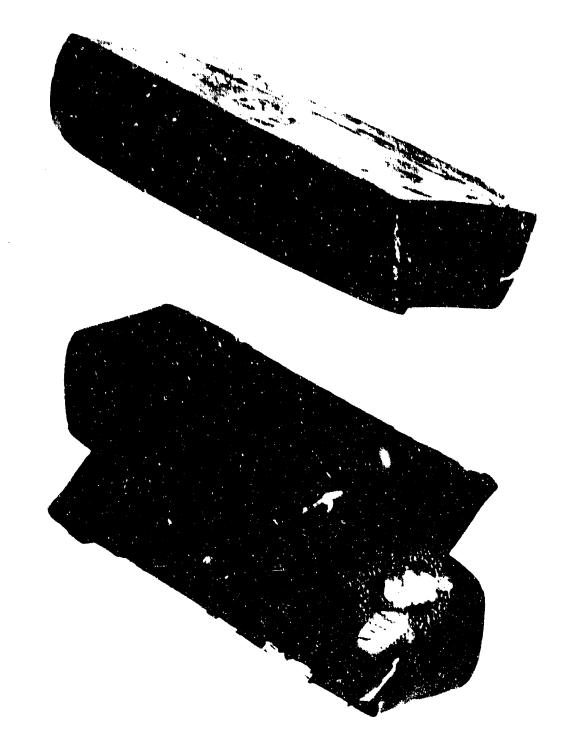


FIGURE 2. HOT WORKING STUDIES

FIGURE 3. EXAMPLES OF FORGED REDUCTIONS OF 1-605 BAR STOCK



FICURE 4. EXAMPLES OF L-605 BARS WHICH CRACKED DURING FUNCING.

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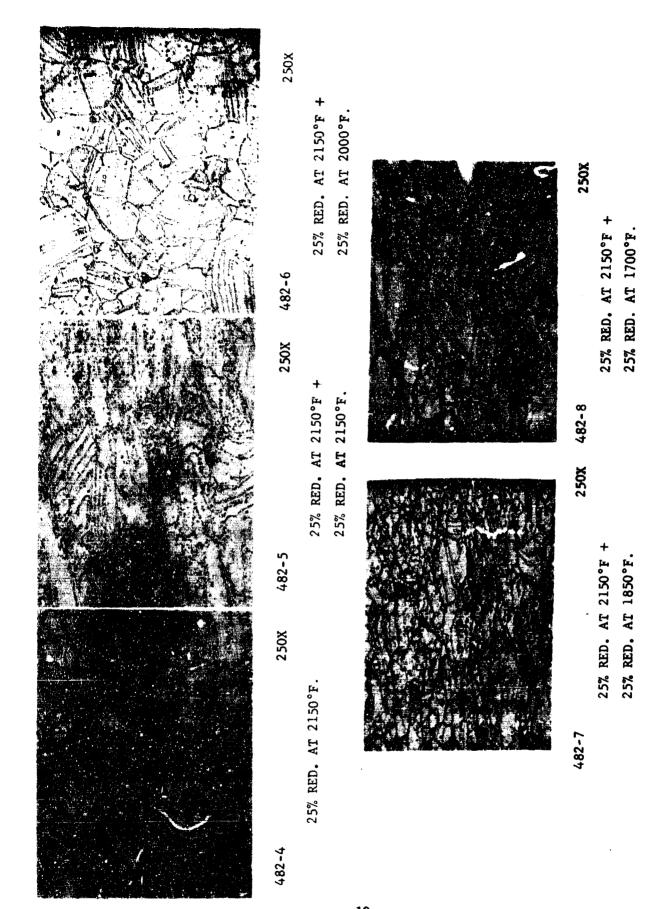


FIGURE 5. EFFECT OF HOT WORKING ON MICROSTRUCTURE OF L-605.

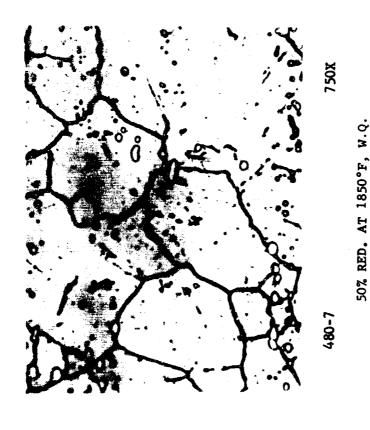


FIGURE 6. EFFECT OF LOW TEMPERATURE HOT WORKING ON THE MICROSTRUCTURE OF L-605

solution by proper thermal treatments, it would be desirable to minimize this condition during forging, thus precluding the need for an unnecessarily high temperature solution heat treatment which could cause excessive grain growth. Based on microstructure, the optimum final forging temperature appears to be near 2150°F.

To investigate effects of composition, melting process, and hot working parameters on mechanical properties, particularly ductility and hardness, round tensile specimens (0.190 diameter x 1 in. guage length) were machined from each sample and tested at room temperature using a strain rate of 0.005 in/in/min. to yield and 0.05 in/in/min. to ultimate. The data are summarized in Tables III and IV. Table IV also includes hardness readings which were taken in triplicate and averaged. The data showed an expected general increase in strength and hardness with a corresponding decrease in ductility as the forging temperature was lowered in all heats. The data were comparable for all heats with the exception of No. 3 (the high silicon heat) which was generally of higher strength and lower ductility. The tensile data are also of value for producing other L-605 forged parts to be used in the as forged condition where higher than annealed properties are required, eg., the SCP/LASRM combustion chamber dome (a hemispherical shaped forging).

3. COLD WORKING STUDIES

To investigate effects of composition, melting process, and annealing treatments on cold working characteristics of L-605, cold working studies were performed by rolling specimens from each heat (reducing the 1 1/4 in. dimension) using ~25 and ~40% multiple reductions with intermediate annealing treatments of 1 hr. at 2000, 2150, and 2250°F followed by a water quench. The cold rolling was performed at Aeronutronic Division of Philoo-Ford, Newport Beach, California. A qualitative summary of the cold workability of each heat is shown in Figure 7. The No. 3 heat (high silicon) was noticeably the most difficult to work. Heats No. 4 and 6 with the very low silicon (0.04% max.) were essentially the same as the other heats, i.e., Nos. 1, 2, and 5 which contained 0.25% Si max.

TABLE III

	Condition	Hon		Room Temperature Tensile Properties of Hot Worked L-605 Bar	erature	Tensile	Proper	rties of	Hot Wol	ked L-6	05 Bar				
Forging		Reforg.			o	0.2% Y.S.	- KSI					UTS	- KSI		
Temp	Red.	Temp.													
(°F)	(%)	(•F)	(%)	Heat#1	2	3	4	5	9	1	2	3	4	2	9
2150	25			86.7	93.9	103.1	94, 1	95.6	82.2	164, 7	167.1	177.4	159.7	161.8	150.0
2150	25	2150	25	113, 3	92.3	107.2	111.5	97.2	88.8	177,6	164, 6	177.7	162.9	160.4	167.4
2150	25	2000	25	123.5	121,0	119.6	103.7	119,3	114,0	182. 4	179.0	182.6	164.7	172, 7	172.1
2150	25	1850	25		128.2	133.9	117.0	123.1	136.3	166.7	175.2	184.6	160.7	170,9	178.4
2150	25	1700	25	146.1	155.7	138.4	128.4	123.7	148.8	182.6	194.6	179.3	167.1	166, 6	181.8
2000	25			ı	112.2	109.8	104.6	107.2	86.4		172.0	178.5	154.8	166.4	151.0
2000	25	2000	22	114.6	106.5	109.6	94.9	103.0	107.5	180.7	168.3	181, 4	160.3	165.8	167.2
2000	25	1850	25	ċ	122.3	130.8	119.0	129.5	142.0	172.7	168.2	179.9	166.3	169.8	178.7
2000	22	1700	25	138.2	138.3	132.8	125.8	143.8	141.0	174.5	173.3	176.2	165.4	177.5	180.1
1850	25			142.8	137.5	160.7	121.7	134.9	99. 7	181.6	178.5	193, 3	159.5	174.7	152.6
1850	25	1850	22	147.3	138.3	140.6	135.9	126.8	136.2	185.4	177.9	184.3	180.8	176.9	174.5
1850	22	1700	52	145,5	165, 7	177.6	130.0	150.0	150.4	179.9	194, 2	211.9	165.1	181,9	185.7
1700	22			136.4	144.0	158.9	135.4	135.0	106.8	172.2	181.9	193, 3	165.0	172.3	158.4
1700	25	1700	25	131.5	139.2	157.5	140.5	131.3	177.3	169.2	175.0	184.4	168.6	171.9	206.5
2150	20			95.5	88.8	101.2	86.7	89.6	79.5	171.8	166,5	176.7	155.5	162, 7	161.4
2150	20	2150	20	103.2	109.3	126.6	101.4	103.3	113.0	171.8	178.5	184.3	166.2	174.3	182.9
2150	20	2000	20	154.7	137.0	163.1	151.1	151.5	135.2	197,3	187.3	201.5	187.3	192, 1	189.9
2150	20	1850	20		189, 2	152.2	175.2	187.8	156.0	218.2	215.4	198.4	201.2	211.6	196, 1
2150	20	1700	20	141.7	195.4	145.5	200.8	218.3	187.5	190, 1	223.5	193, 3	222.0	239, 9	214.8
2000	20			ċ	97,1	118.2	100.3	96.0	95.8	161.0	168.3	182.2	165.2	164.1	164,4
2000	20	2000	20	144.5	149.9	189.6	119,3	127.7	135, 7	192, 7	196.4	217.6	177.5	181.0	193.2
2000	20	1850	20	183.0		163.8	181.4	195.3	162.7	214.8	ı	201.4	207.0	223, 3	202.5
2000	20	1700	20	185.3	204.1	202.6	201.0	212.3	203.4	228.9	233, 1	229.4	226.9	233.6	226.6
1850	20			130.8	130.0	151.4	121.7	134.5	145.1	178.8	176.4	192,1	168,1	134, 7	185,5
1850	20	1850	20	165.1	159, 1	171.8	175.2	149.6	163,6	204.0	201,3	211.0	205,8	194.4	201.1
1850	20	1700	20	212.5	221, 2	ı	203.3	211.5	205.6	242.3	246.0	ı	231, 5	237.7	233.4
1700	20			152.1	150.1	1	143.0	135.6	141.6	197.9	137.1	1	175.7	170.4	183,1
1700	20	1700	50	168.7	210.5	170.6	205.4	200.3	193.2	205.7	237.3	204.9	229.7	224.0	218.9

Note: All tests rur in duplicate

TABLE IV

Room Temperature Ductility and Hardness of Hot Worked L-605 Bar

	200	Condition													\lceil
Forging		Reford				; ;	! }								
Temp	. Red.					("I) TE %) - % RA *				Ï	ardn	Hardness-	Rc**	*
(°F)	i	- 1	(%)	Heat # 1	2	က	4	5	9		83	က	4	വ	9
2150	25			50.5/45.2	49.0/37.3	35.0/35.2	48.0/41.6	46.5/40,9	42.5/33.7	34	31	36	31	31	30
2150	25	2150	25	41.0/47.9	47.5/40.6	36.5/30.0	42.5/40.9	45.0/41.0	50.0/38.5	40	33	33	35	32	31
2150	25	2000	25	35.0/32.2	41.0/39.6	26.5/24.3	40.0/42.7	36.5/30.9	32.5/27.5	43	40	35	34	40	36
2150	22	1850	22	31.0/31.4	28.0/26.3	25.0/22.5	37.0/37.0	34.0/32.6	18.0/18.0	42	38	36	41	41	40
2150	25	1700	22	19.5/26.5	22.5/26.5	11.0/17.7	36.0/38.8	12.0/30.0	16.0/17.7	46	44		1	47	41
2000	25			1	36.5/31.9	27.5/26.1	39, 5/36, 6	36.5/33.0	37.0/31.0		35	က	31	33	28
2000	25	2000	25	42.0/34.2	32.0/30.7	24.5/20.1	42.0/34.7	44.5/39.8	34. 0/29. 2	30	38	41	33	31	32
2000	25	1850	25	24.0/24.3	17.5/19.7	18.0/17.8	34.5/32.3	32.5/32.1	16.5/18.3	43	40	45	42	36	39
2000	25	1700	25	19.5/24.6	21.0/23.8	13.0/15.8	31.0/30.7	26.5/25.6	15.5/16.1	45	45	35	34	36	39
1850	25			23.5/27.5	30.0/26.0	13.5/22.5	33.5/39.8	32.0/34.1	24.0/21.0	36	26	41	36	38	33
1850	25	1850	25	17.5/18.5	21.0/20.7	14, 5/14, 4	35.0/37.7	36.0/28.7	13.0/13.9	37	40	34		38	41
1850	25	1700	25	19.5/21.3	13.0/25.9	8.0/11.5	34.0/38.9	13.0/16.1	12.0/12.9	38	43	38		42	45
1700	25			0	28.5/31.0	18.5/22.9	34.0/36.6	34.0/33.6	32, 5/28, 7	35	44	44	37	38	34
1700	25	1700	25	26.0/32.2	17.5/26.4	8.0/8.9	28.5/28.2	30.5/26.5	9.0/13.8	39	34	39		40	46
2150	20			5/49.	44.0/33.8	41.5/36.6	47, 5/40.0	51.5/39.9	53.0/44.0	35	30	40	29	33	34
2150	20	2150	20	49.0/54.5	51.0/53.6	37.0/37.4	51.0/45.8	53.0/48.3	47.5/45.2	34	20	40	33	37	42
2150	20	2000	20	29.0/44.2	39.0/39.3	25.0/26.5	35.0/43.9	39.5/47.8	27.5/26.5	46	45	42	45	42	37
2150	20	1850	20	0/34.	13.0/38.6	25, 3/27, 3	19.0/45.8	5/42.	16.5/19.2	41	49	46	45	46	45
2150	20	1700	20	31.0/36.4	9.0/26.8	21.5/22.0	12.0/45.8	9.0/37.5	7.5/20.5	46	49	46	46	48	48
2000	20			37.5/35.8	41.5/32.8	30.0/29.0	42.0/42.8	36, 5/37, 9	36.5/31.7	31	36	40	37	34	48
2000	20	2000	20	32.0/38.8	36.5/45.3	7.0/28.2	42.0/46.7	43.5/42.1	31, 5/29, 6	41	45	49	36	34	40
2000	20	1850	20	11.0/31.2		24, 5/32, 6	23.0/44.1	13.5/44.9	18.5 21.1	48	46	46	46	44	46
2000	20	1700	20	10.0/42.0	8.0/28.4	7.5/20.0	9.0/42.3	10.0/40.7	5/26.	20	46	49	46	42	46
1850	20			22.0/29.7	24.5/21.8	20.0/22.0	33.0/34.0	26.5/37.9	14.5/16.9	42	33	42	<u>ლ</u>	43	21
1850	20	1850	20	17.0/33.8	26.5/32.4	15.0/36.0	16.0/38.7	31.5/35.4	20.0/24.8	47	42	20	46	44	47
1850	20	1700	20	7.5/29.6	7.5/32.6	1	10.5/41.4	11.5/36.0	5.0/16.8	30	37	1	46	46	50
1700	20			18.0/34.3	26.0/31.0	•	32.5/30.4	31.0/38.7	28.0/27.1	43	41	•	39	47	42
1700	20	1700	20	15.5/25.0	11.0/38.8	10.0/19.0	10.0/37.8	13.0/36.7	9.3/26.1	46	4	43	52	20	50
* 4	ore of	# Among of duplicate toneile requite	onello	mognite											1

*Average of duplicate tensile results. **Averages of three readings.

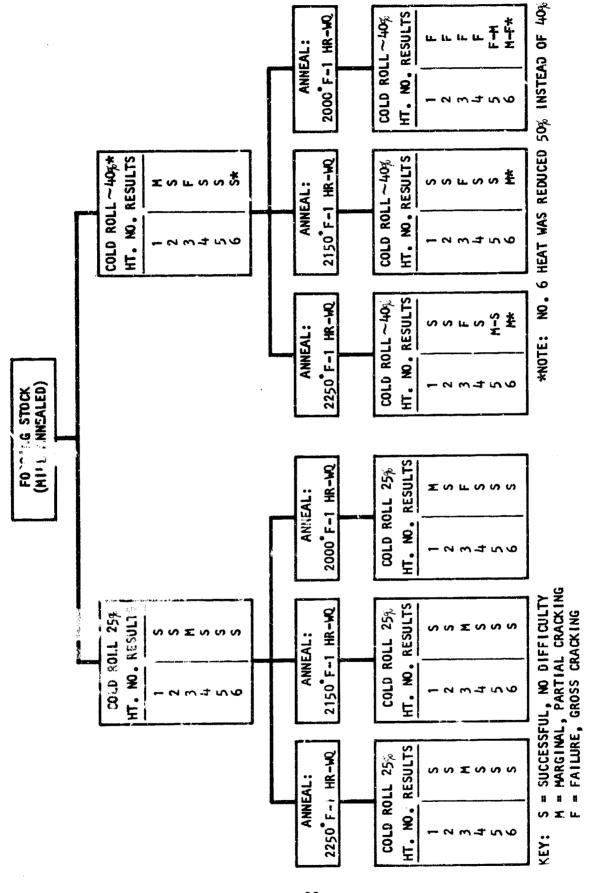


FIGURE 7. COLD WORKING DATA

The air melted heat (No. 6), was reduced 50% instead of 40%, which resulted in additional material loss due to cracking. This difference in reduction resulted from the fact that the 1 in. x 1 1/4 in. bar stock from this heat was processed first according to plan; the bar from the remaining heats (3/4 in. x 1 1/4 in.) twisted badly making reduction past 40% impossible. Due to the limited capacity of the rolling mill used and the geometry of the L-605 bars, considerable work hardening of the surface occurred during the multiple passes required to achieve ~25 or ~40% reductions. This caused more difficulty than would be expected during shear spinning where these reductions can be accomplished in a single pass. As indicated in Figure 7, the 2000°F treatment was insufficient for the subsequent reductions compared to the higher temperature (2250°F and 2150°F) solution anneals.

Tensile specimens were machined from samples of each material processing variable and tested at room temperature. These results along with hardness measurements are summarized in Tables V & VI. The blank spaces in the tables indicate lack of sufficient material for testing due to excessive cracking during cold rolling. This problem was most severe for the high silicon (No. 3) heat. Based on the mechanical properties, hardness data, and workability (Figure 7), complete annealing did not occur at 2000°F, but at 2150 and 2250°F annealed properties were achieved. No well defined effects of composition were noted, except for an indication that the high silicon heat (No. 3) was slightly stronger and less ductile than the others.

The 25% cold worked specimens did not consistently reach the SCP/LASRM design target of 175 KSI yield strength which could have resulted from machining the specimens from varying locations within the work hardened bar. The aging treatments (700°F and 1200°F) increased the yield strengths significantly over this figure. A detailed discussion of the aging studies is included in Section III-4. The microstructures of cold worked material annealed at 2000, 2150, and 2250°F are shown in Figure 8. The 2000°F photomicrograph shows partial recrystallization with

TABLE V

Room Temperature Tensile Properties of Cold Worked, Annealed, and Aged L-605 Bar

		•				` 						
Condition		o.	0. Z% Y.S.	- KSI						- K3I		
Hes	Heat #1	81	က	4	r.	9	-	81	က	4	ເດ	9
25% Cold Work	182.6*	182.9	164.0	146.0	123.3	147.1	216.2*	219.6	197.5	180.4	185.9	191.3
25% CW+2250'F Anneal	62.6	65.3	65.1	63. 4	59.2*	59.4	142.8	150.7	152.0	146.2	142, 2*	138.6
25% CW+2150°F Anneal	72.7	64.8*	78.4	66.0	62.6	61.8	147.4	151.7*	157.9	141, 2	139.0	
25% CW+2000'F Anneal	91.5*	76.9	92. 7	83.6*	85.0*	82.5	170.1*	170.1	178.7	166.6*	167.2*	173.2
25% CW+2250°F+25% CW	183.9	177. 4	169.7	162.5	173. 1	174, 3	217.3	232. 7	237.8	217.6	214.4	205.9
25% CW+2150°F+25% CW	180.6	188.8	184.9	172.7	172.1	191.2	238.2	232.6	257.7	211.6	223.5	238.5
25% CW+2000 F+25% CW	193.6	196. 7	192.2	185.0	205.4	198.6	259.1	253.3	257.7	243.1	245.7	250.0
25% CW+2250°F+259, CW+2150°F	69.5	59.6*	76.6	60.7	60.8	63. 7*	161.1	140.8*	162.9	142.2	140.8	143.3*
25% CW+2250 F+25% CW+ 730 F-10 hr 218.	218.6	235.8	•	190.0		190.6	229.0	249.7	•	209.3		206.6
25% CW+2250 F+25% CW+1200 F-1 hr	237.7	227.1	228.1	204.8	227.1	2007	250.9	246.8	255.8	221.6	239.9	214.5
CW+2150 F+259	63. 1	64.5	73.2	60.2	64.0*	65.2	149.1	149.4	157.3	141.5	138.1*	147.5
	238.7	226.8	,	203.5	•	229.7	254.0	241.5	i	217.6	•	247.0
CW+2150° F+259	243.6	228.5	246.6	216.2	224.6	234.3	263.8	251.9	277.3	231.6	242.6	264.4
25% CW+2000 F+25% CW+2150 F	9	67.6	•	60.4	62.6	,	143.6	148.4	•	139.5	142.5	
25% CW+2000°F+25% CW+1200°F-1 hr	249.5	257.3	ŧ	254.8	254. 4	247.2	280.7	281.4	•	272.7	276.3	277.8
~40% Cold Work	198.7	207.7	205.6*	150.9	163. 1	202.1	242.0	250.7	248.9*	238.2	258.5	239.9
~40% CW+2250°F Anneal	63.3	65.6		67.5	62.0	64.8	138.1	144.0	•	135.7	141.2	134.4
~ 40% CW+2150°F Anneal	70.4	83.2	•	61.8	65.4		148.8	160.8	•	135.3	140.9	
~40% CW+2000°F Anneal	94.0*	102.9	•	88.8*	101.4	93.9	167.3*	175.8		163.0*	169.2	174.1
~40% CW+2250°F+ ~40% CW	179.6	•	•	167.9*	174.3	203.5	248.6	•	•	259.6*	251.4	248.2
~40% CW+2150°F+ ~40% CW	198.5	183, 7	•	187.1	193.9	,	269.3	289.1	ŧ	226.8	255.3	•
~40% CW+2250°F+~40%+2150°F	63.8	68.3	•	61.9	62.3	•	140.9	144,4	•	139.6	140.9	,
~40% CW+2250 F+ ~40%+700 F-10 hr	238.4	ı		232.8	•		256, 6	•	•	256.6	•	248.8
~40% CW+2250°F+~40%+1200°F-1 hr	237.2	•	•	249.5	247.5	243.2	265.8	•		252, 3	243.2	247.0
~40% CW+2150°F+~40%+2150°F	ιο.	73.5	•	59.5	65, 4*	,	146.5	160.9		143.3	141.4*	,
~40% CW+2150°F+~40%+700°F-10 hr	247.2	•	•	250.0	•		282. 4	249.1		256, 6	1	,
40% CW+2150°F+~40%+1200°F-1 hr	269. 1	276.1	•	235.6	259. 2	•	281.5	296.7	ı	253.2	261.3	,

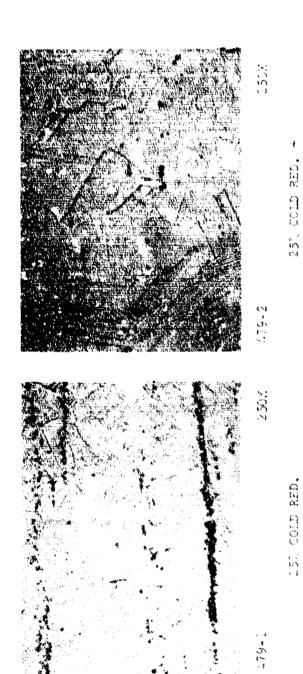
Note: All tests run in duplicate except those shown by * which are singular

TABLE VI

Room Temperature Ductility and Hardness of Cold Worked, Annealed, and Aged L-605 Bar

<u> </u>	Condition			Q. EL.(1")	1 - 9 RA***				Hardness	nest	-R	*	Γ
		Heat #1	8	6		ប	9		8	ဆ	4	ပင္မ	9
	25% Cold Work	9,0/33,5*	6.0/15.4	20.0/24.0	26.4/37.4	13,0/36,3	21.0/30.6	41	38	46	43	31	45
NAC 188	25% CW+2250°F Anneal	73.0/47.9	65.5/45.8	67.0/45.0	67.0/47.6	60.0/39.5	74.5/46.2	21	21	31	19	22	21
	25% CW+2150°F Anneal	62.0/53.7	67.0/46.7	56.0/42.2	66.0/54.5	64.0/49.7	71.5/47.6	29	24	31	21	13	19
	25% CW+2000°F Anneal	55.0/62.5	50.0/37.7	50.5/37.9	53.0/45.0	59.0/52.3	48.0/38.3	36	34	35	30	30	34
	25% CW+2250° F+25% CW	10, 5/33, 9	5.5/13.7	8.5/23.8	5.0/11.9	7.5/11.9	9, 5/24, 7	43	45	43	43	43	43
	25% CW+2150°F+25% CW	7.5/13.7	6.5/20.8	7.9/20.9	6.0/14.8	5.0/10.0	8.0/15.5	47	45	46	41	44	43
	25% CW+2000°F+25% CW	9.5/52.9	5.5/21.6	5.5/16.2	3.0/24.9	7.0/14.8	5.0/11.6	47	44	46	43	43	50
	CW+2250°F+25% CW+2150°F	59.5/52.2	68.0/46.3	59.0/51.9	66.5/48.7	64.5/43.4	68.0/52.6*	17	21	31	16	19	17
	25% CW+2250 F+25% CW+700 F-10 Ir 10.0,	೯	3.0/11.0	•	3.5/19.0	•	9,0/27.0	47	43		44	•	46
	25% CW+2250°F+25% CW+1200°F-1hr 2.5,	2.5/ 7.0	1.0/2.0	1.0/2.5	2.5/2.5	1.0/2.0	2.3/8.0	17	42	51	47	48	48
	CW+2150°F+25% CW+2150°F	66.5/49.3	66.5/51.6	59.5/46.2	68.5/46.4	66.0/50.5	65.0/49.4	19	20	56	21	18	20
		10.0/28.0	7.0/26.0	,	5.0/16.0	•	7.0/21.0	45	41	ı	47	•	51
	25% CW+2150°F+25% CW+1200°F-4 hr 2.0,	2.0/ 7.0	1.0/2.5	1.0/3.0	2.0/4.0	1.0/ 1.5	2.3/3.0	48	49	52	20	50	52
29	25% CW+2000°F+25% CW-2150°F	67.5/60.2	62.5/50.1	•	64, 0/43, 8	67.0/49.3	1	21	21		19	20	1
··	25% CW+2000°F+25% CW+1200°F+1hr 1.0,	1.0/12.0	2.0/5.0	•	2.0/5.0	1.0/2.0	1.5/2.0	51	49	1	52	51	53
	~40% Cold Work	5.0/16.6	3.5/21.4	3.0/9.5*	4.0/5.3	4.0/13.0	7, 5, 12, 4	39	46	45	43	44	1-
	~40g しW+225プF Anneal	69.5/59.4	69.0/26.0	1	65.0/51.2	62.0/46.3	73.5/56.0	20	21	•	19	50	16
	CW+2150'F Anneal	59,		ı			60.0/52.7	21	24		18	21	21
	~40% CW+2600° F Anneal	50.0/53.2*	47.5/46.4	ı	52.0/56.7*	47.0/48.4	41.5/36.5	31	36		31	36	37
	~①季 CW+2250°F+~40% CW	5.0/6.3	•	•	3.0/2.1	6.0/3.2	3.0/5.3	45	•	1	4-	46	50
-	~40% CW+2150°F+~40% CW	3.5/8.3	4.0/5.8	ı	1.3/2.7	4.5/4.2	1	39	52	ı	45	47	•
(~40% CW+2250°F+~40%+2150°F	66.0/62.0	68.0/57.4	ŧ	64.0/55.1	65.0/51.0	•	20	24	ı	20	18	1
	~40% CW+2250 F+~40%+700 F 40 hr	2.0/10.0	•	•	1.0/8.0	•	4.5/6.0	42	ı	1	48	1	51
	~40% CW+2250°F+~40%+1200°F - 1hr	1.0/1.0	1		0.5/1.0	0.5/ 1.0	1.0/ 1.0	51		1	52	51	52
(8.09/0.99	59.5/57.0	ı	63.0/42.6	65.0/52.3	ı	23	28	•	13	21	1
	CW+2150 F+~408+700 F - 10 hr	3.0/ 7.0	0/ 1.	ı		,	1	52	51	1	48	1	•
لـــــــــــــــــــــــــــــــــــــ	~404 CW+2150 F-~404+1200 F-1 hr	1.0/ 1.0	0.5/ 1.0	•	1.0/ 1.0	0.2/0.0	1	55	52	1	49	20	,
*	** Data are averages of three readings												

Data are averages of three readings *Tests run in duplicate except those shown by * which are singular







25% COLD MED. +

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31

heavily precipitated grain boundaries. Both of the higher temperatures were effective in resolutioning the precipitates. The \$250°F trestment resulted in significating greater grain growth than the \$150°F trestment which illustrates the advantage of minimizing annealing temperature for grain size control of shear spinning preforms.

4. AGING STUDIES

Several investigators (** 5) have shown increases in strength (and an embrit fling tendency) resulting from prolonged exposure of 1. 600 to intermediate temperatures between 700 and 1800°E. Hecause the SCP LASIM design desires 175 KSI yield strength, the possibility of employing a post shoar spinning aging frost ment for increased strength was considered attractive. This would allow greater flexibility in the SCP/LASIM production process in that the strength requirements of the part would not necessarily be entirely dependent upon the smount of cold work schieved during the final shear spinning reduction.

Samples from the cold working study (Section III: 3) were aged after single and multiple cold reductions of ~25 and ~40%. Aging treatments of 700, 1200, 1300, 1650 and 1800° r for periods of 1, 10, and 100 hrs. were selected to isolate an optimum post slear sleaning treatment (if any) for best combinations of strength and ductility. The tensile data (Tables V VIII) were averaged (leaving out the No. 3 high silicon heat) and plotted as shown in Figures 9-14. Two relatively short time aging treatments appeared effective in increasing strength, i.e., 1 hour at 1200°F and to hours at 700°F. The latter treatment showed less reduction in ductility than the former with essentially the same increases in strength.

Because notch sensitivity of materials for such applications as SCP/LASRM center sections is of concern, notched tensile specimens ($k_{\rm t} = 7.8$) were machined functorial in various cold worked and aged conditions. Specimens were randomly selected from heat numers 1, 2, 4, 5, and 6 (omitting number 3, the high silicon heat). The material conditions tested, which are detailed in Table IX, represented 25 and 40% cold rolled material, with and without interstage anneals of 1 hour at 2150°F and 2250°F and post—working aging treatments of 1 hour at 1200°F

TABLE 'T

Boom Lemperature Tensile Properties of Cold Worked and Agrid 1.466 Bar-

7													-
	Condition	.,	2% Y.S.	75					K	KS			
•	A design of consequences of co	Heat #1 2	٠٠,	v#	ω	14 5	. 4	٧,		**	w	(4)	
_	255 CW - Age 1800'E - 1 hr	9.2 115.	94.6		¥.	324.6	\$		4	2	;	3	
	sur 61 -	96. 8 95. 1	i- က တ	•	53. C	433 , 4 479	est est to	113	1:	٠.	1: 1 1: 1 1: 1 1: 1	;;;	
	- 166 hrs.	2.8 \$ 32.		36.3	4) 6) (0)		67 67 47 47	्र क्षे	()) (4) (1)	, Yi	123	13	
	25 CW - Age 1850F - 1 hr	42.2	146.3	132.4	342.9	ສໍ	2.4 f -	ري دو دو دو	Çî Pî Hi	41°	1,8 181 8 - 1 -	٠,٠ بها دو	
	10 110	126.	127, 9	112,5	4	173	4 - -#1		.∉\	11	100	Ŕ	
	- 100 hrs	න ග ශ	က်	97.6	6;1 6;1 1#		10 10 10 10 10 10 10 10 10 10 10 10 10 1	2.5	ت ا ا			ri	
	25 F CW + Age 1.30F - 1 hr -	ú	s S	136.7	139.7	146.9	186	188	- .	7	ιμι (φ) (γ)	.;	
	- 10 hrs	F7				205. 5	क्ष क स्ट		(#)	ų,		** 4	
	~ 100 hrs	125.8 137.8	(2) (4)	130.3	117.6	146. 7	163.65	2.68.2	3.55	₩ 1.* 13.7 17.1	4.837	113	
•	257 CW - Age 1200 F - 1 nr	223. 0 176. 6	268.7	275.6		265.2	् जू ज	64 (3) (3)		0)	1382	64 65 64 64	
	874 01 ·	7 5 3	•	221.8		164.0	3.06	245.2	•	240	61	Š	
	- 160 hrs		241.2	158.3	3.011	ស ស្វា ស្វា ស្វា	235.	•	ख हो है इस	e3 -5 -00 -1	10 .; .;	1 3 3	
	25% CW - Age 700FF - 1 hr	59. 7. 16	161.9	375.6	15 10 10	201.1	*		105 38	- ch	٠.	(n)	
3.1	- 16 hrs		<u>ئ</u>	153.4	74		245.2	121	, ; 103	: 1 ::: ::::	# 5 # #	225	
	- 166 hrs	63. 4. 169.	τη: (ψ	217.9	183	3		;;	un un	3	ું આ	
	-40% CW + Age 1800F - 1 hr		•	-41	•	10 120 110	1	L S		, ti	•	41 ⁴	
	+	9	,	(X)	64.	38		4	,	14			
	- 106 hrs.		ı	13	$\dot{\mathbf{z}}$	4.12	Ţ.	•	•	159.5		S (S)	
	40 € CW + Age 1656 F - 1 hr	42. 7. 14	•	125.5	128.2	196.1	ល់ ស៊ី មា		,	(g) (g) (g) (g)	ক নে জ	222	
	- 16 hrs	117.6 117.1		139.2	115,4	348. E	0.58E		,	(5) (3) (3) (4)		: 45.	
	- 100 hrs	- 103.8	1	101.1	103.0	105.3	•	10 13 14 14	•	60 60 10 10	2.5	(B)	
	-40% CW + Age 1350'F - 1 hr	70.3 157.	•	40.	60 60 60 60	194. 7	i D	1 - 31	•	69 10 36 13	(A) (A) (A) (A)	d	
	- 10 hrs	263, 8, 226, 5		218, 1	4	205.9	222. 6	268.3	•	Ş	•	0 0 0 0 0 0 0	
	- 100 hrs	32.4 122.		20.	132.4		(- ()	3.5	•	(5 (0)	200.4	4	
	-40% CW + Age 1200F - 1 hr	4	1	٠	, 0.	228.1	270.1	266.9	•	226.3	57.7	254. 8	
	- 10 hrs	ŧ	•	t	233. 9	5.2	•		•	١	in.	(S)	
****	- 100 hrs	228.9		218, 3	•	•	254.2		,	242.8	186, 1	ઝુ	
	-40% CW + Age 700°F - 1 hr	214, 2 219, 2	,	220.8	205, 6	αň	253, 7	į -	•		251.8	250.6	
	- 10 hrs	38.6 240.		αñ	-	93.2		9	•	£3.	62	23.	
	- 100 hrs	6.3		233. €	239. 7	1, 3	276.6	250.5	•	277. 1	266.5	(- -	
						!							ĺ

Note: Data are result of single tests.

Room Temperature Ductility and Hardness of Cold Worked and Aged L.-605 Bar

ondition.			9 57	/1113 - 02 DA#	*V			Hay	Hardnose	1	**Ja	j''-
COMMINION			5	- (+)				1191		1	}	
	Heat # 1	2	co	4	5	ê		~	ت ا	4	ت.	9
25% CW + Age 1800°F - 1 hr	28.0/13.9	35.0/23.7	42.0/31.7	47.0/37.0	44. n/29. s	19. 0/14. 4	35	38	37	34 3	35 4	41
- 10 hrs	34.0	42.0/38.7	34.0/32.5	41.0/37.7	35.0/39.4	37.0/36.0	38	33	39	36 3	30 3	34
- 100 hrs	31.0/	46.0/32.4	38.0/28.2	38.0/28.2	36.0/26.5	31.0/23.5	35	33	36	34 3	33 3	34
25% CW + Age 1650°F - 1 hr	28.0/23.7	28.0/20.9	16.0/16.0	26.0/28.7	23.0/24.7	6.0/8.4	42	38	4	36 3	38 4	4
1		22.0/20.0	18.0/16.1	35.0/26.6	36.0/27.5	11.0/12.2	36	38	43	37 3	33 4	41
- 100 hrs	23. (26.0/21.0	26.0/19.0	29.0/25.4	33,0/26.5	22.0/14.3	38	38	41	36 3	38 4	40
25% CW + Age 1350°F - 1 hr	16.0/18.9	2.0/ 7.3	2.0/6.2	31.0/29.6	18.0/17.9	5.0/8.3	37	46	40	40 3	32 3	39
ı	9.0/12.2	•	3.0/14.4	3.0/9.3	,	1.0/ 1.1	36	•	47	45	4	46
- 100 hrs	12.0/10.5	12.0/16.0	15.0/16.2	15.0/15.3	19.0/17.3	11.0/19.0	35	44	46	38 4	42 4	- 9
25% CW + Age 1200 F - 1 hr	2.0/ 6.4	17.0/21.0	2.0/6.4	2.0/6.4	2.0/8.5	3.0/ 7.4	3	47	46	41 4	44 4	49
- 10 hrs		1.0/2.1	8	1.0/2.1	1.0/3.1	2.0/ 5.4	43	49	1	47 4	43 4	48
- 100 hrs	16.0/ 4.2	a	1.0/ 3.0	9,0/11,6	5.0/6.3	1.0/ 1.0	48		53	41 4	44 5	51
25% CW + Age 700°F - ihr	24.0/37.7	25.0/31.6	12.0/26.6	5,0/18,1	28,0/30,3	5.0/6.4	46	43	44	40 3	39 3	34
- 1	3.0/	6.0/11.3	2.0/12.4	16.0/27.2	23.0/28.2	40.0/10.6	43	45	45	34 4	44. 4	48
- 100 hrs	31.0/37.7	31.0/34.8	50.0/19.2	14.0/43.9	4.0/33.4	32.0/40.7	38	42	20	38 4	43 4	40
1400 CW + Amo 1800 E = 1 hr	37 0/97 3	40 0/31 0	1	44 0/34 5	I	17 0/15 0	40	90	,	90	_	5
ı	34	39 0/38 1	•	38 0/37 5	35 0/38 1		2 6	, E				3 8
	38.0/31		(33 0/26 4			2 4	3			cc	3 %
	00.07	•	•	0/ 70	0/ 70	·74.	2					 >
-40% CW + Age 1650°F - 1 hr	21.0/23.	14.0/11.3	•	ı	38.0/29.9	÷	43	4 3				8
- 10 hrs	23.0/18.3		•	20.0/23/6	25.0/18.8	14.0/16.0	41	36				43
- 100 hrs	,	35.0/28.3	•	30.0/28.7	33.0/29.2	25.0/19.0	37	40	,	34	38	51
-40% CW + Age 1350°F - 1 hr	6.0/13.1	13.0/14.1	•	16.0/22.5	8.0/13.2	1.0/ 4.1	45	43	1	34 4	41 4	47
- 10 hrs	1.0/	2.0/3.1	ı	2.0/5.2	•	1.0/ 1.0	40	49	•	47	LC)	51
- 100 hrs		23.0/22.9		12.0/13.1	22.0/19.8	10.0/8.4	45	42	,	42	43 4	45
~40% CW + Age 1200°F - 1 hr	/ 3.2	1.0/3.3		ı	က	1.5/2,3	41	51		31 2	25 5	51
- 10 hrs	1	1.0/ 3.2	•	•	1.0/1.1	1.0/2.0	1	49		50 4	49	51
- 100 hrs	1.0/1.1	•	•	3.0/ 4.0	•	1	44	21		47		52
-40% CW + Age 700°F - 1 hr		4, 0/13, 4	•	2.0/3.2	6	4	42	44	1	46	3,	51
	8	1.0/6.2	•		2.4/5.3	4.0/ 7.2	43	45			49	3
- 106 hrs	3.0/16.	જાં	•	3.0/5.3	0/12.	5.0/17.1	47	46		48		<u></u>

* Data are result of single tests **Data are averages of three readings

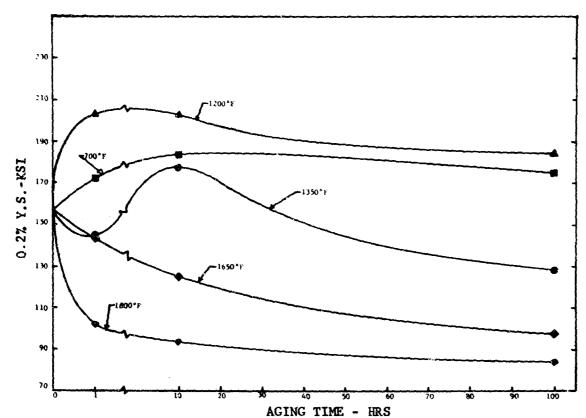


FIGURE 9. EFFECT OF AGING TREATMENTS ON THE YIELD STRENGTH OF 25% COLD WORKED L-605 BAR (ROOM TEMPERATURE DATA; AVERAGES OF HEATS 1, 2, 4, 5, 6)

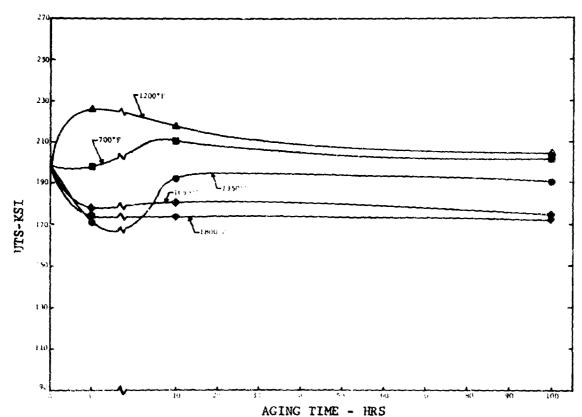


FIGURE 10. EFFECT OF AGING TREATMENTS ON THE ULTIMATE TENSILE STRENGTH of 25% COLD WORKED L-605 BAR (ROOM TEMPERATURE DATA; AVERAGES OF HEATS 1, 2, 4, 5, 6)

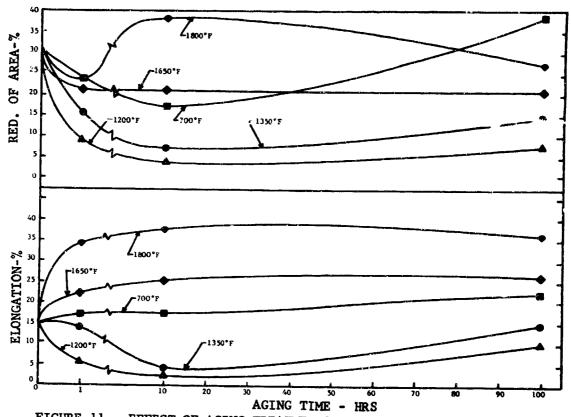


FIGURE 11.. EFFECT OF AGING TREATMENTS ON THE DUCTILITY OF 25% COLD WORKED L-605 BAR (ROOM TEMPERATURE DATA; AVERAGES OF HEATS 1, 2, 4, 5, 6)

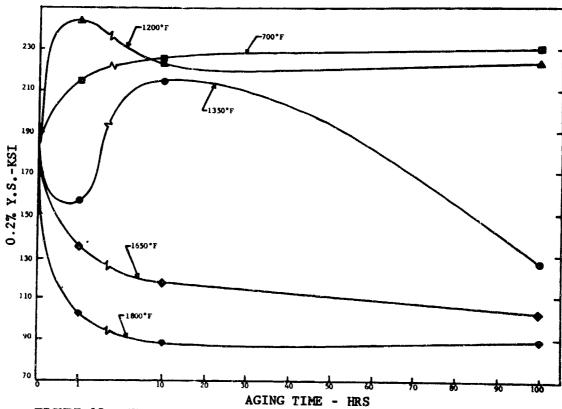


FIGURE 12. EFFECT OF AGING TREATMENTS ON THE YIELD STRENGTH OF 40% COLD WORKED L-605 BAR (ROOM TEMPERATURE DATA; AVERAGES OF HEATS 1, 2, 4, 5)

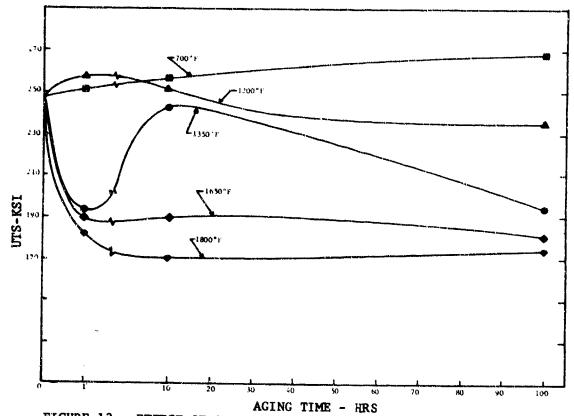


FIGURE 13. EFFECT OF AGING TREATMENTS ON THE ULTIMATE TENSILE ST ENGTH OF 40% COLD WORKED L-605 BAR (ROOM TEMPERATURE DATA; AVERAGES OF HEATS 1, 2, 4, 5)

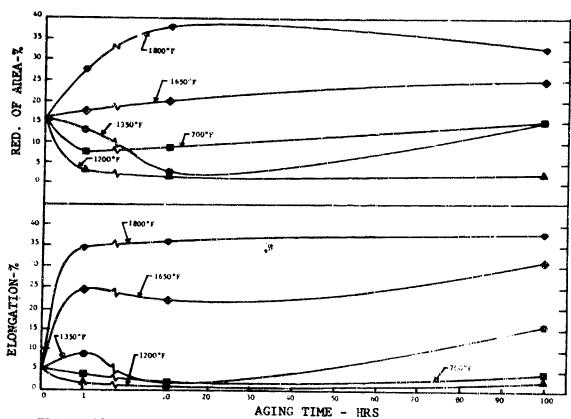


FIGURE 14. EFFECT OF AGING TREATMENTS ON THE DUCTILITY OF 40% COLD WORKED L-605 BAR (ROOM TEMPERATURE DATA; AVERAGES OF HEATS 1, 2, 4, 5)

TABLE IX

NOTCHED TENSILE STRENGTHS OF COLD WORKED AND COLD WORKED PLUS AGED L-605 BAR

CONDITION		NTS* (KSI)	uts (KSI)	RATIO (NTS/UTS)
25% C.W.		326.8***	185.9	1.76
25% C.W. + 2250°F-1 hrW.Q. + 25% C.W.	C. 15.	294.6	214.4	1.37
25% C.W. + 2150°F-1 hrW.Q. + 25% C.W.	G. 55.	299.2	223.5	1 32
25% C.W. + 2150°F-1 hrW.Q. + 25% C.W. + 700°F-10 hrs.	C.W. + 700°F~10 hrs.	324.4	244.3	1.33
25% C.W. + 2150°F-1 hrW.Q. + 25% C.W. + 1200°F-1 hr.	C.W. + 1200°F-1 hr.	240 . 1	247.7	0.97
		306.5	258.5	1.19
407, C.W. + 2250°F-1 hrW.Q. + 40% C.W.	;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;	253.4	251.4	1.01
40% C.W. + 2150°F-1 hrW.Q. + 40% C.W.	C. E.	236.6	255.3	0.93
40%, C.W. + 2150°F-1 hrW.Q. + 40% (C.W. + 700°F-10 hr.	267.4	249.1	1.07
407, C.W. + 2150°F-1 hrW.Q. + 407, C.W. + 1200°F-1 hr.	C.W. + 1200*F-1 hr.	166.7	296.7	0.56

Average of two tests, $K_{\rm t} = 7-8$.

Average of two tests from same heats.

^{***} Single test only.

and 10 hours at 700°F. The data showed that the material retains low notch sensitivity even after 40% cold work, based on notched to unnotched ultimate strength ratios of 0.93 to 1.76 for the various material conditions. However, the 1200°F, 1 hour aging treatment increased the notch sensitivity of the material significantly more than the 700°F, 10 hour treatment. Specimens cold rolled 40% and aged at 1200°I' for 1 hour showed a notched to unnotched ratio of 0.56 which would be considered very low. This supports the tensile data discussed above which showed that the lower temperature aging treatment was more effective in raising the strength of cold worked material without significant lowering of ductility. Based on these data, the 700°F, 10 hour aging treatment was selected for aging the SCP/LASRM shear spun parts.

Selected typical photomicrographs of aged material are shown in Figure 15. It is interesting to note that no precipitate is visible in the 700°F aged material using optical metallography and standard preparation techniques. X-ray diffraction analysis of the aged microstructures is detailed in Section III-5.

5. X-RAY DIFFRACTION STUDIES

As previously discussed, several investigators have shown that a major cause of the embrittlement phenomenon observed during the aging of L-605 in the temperature range 1200°F to 2000°F is the precipitation of a Laves phase along slip planes. The grain boundary precipitation of complex carbides (M₂₃C₆ and M₆C) are also a contributing factor in the loss of ductility. The microstructures (Figures 5, 6, 8 and 15) of samples representing various hot and cold worked conditions with subsequent heat treatments show considerable variation in the quantity, location and appearance of the precipitates. In order to quantitatively establish the types of precipitates formed and their relationship to the mechanical properties presented in Section III-2, 3, & 4, the precipitate phases from eleven specimens from heat 5 (Table I) in the conditions shown in Table X were extracted by electrochemical methods and identified by x-ray diffraction analysis.

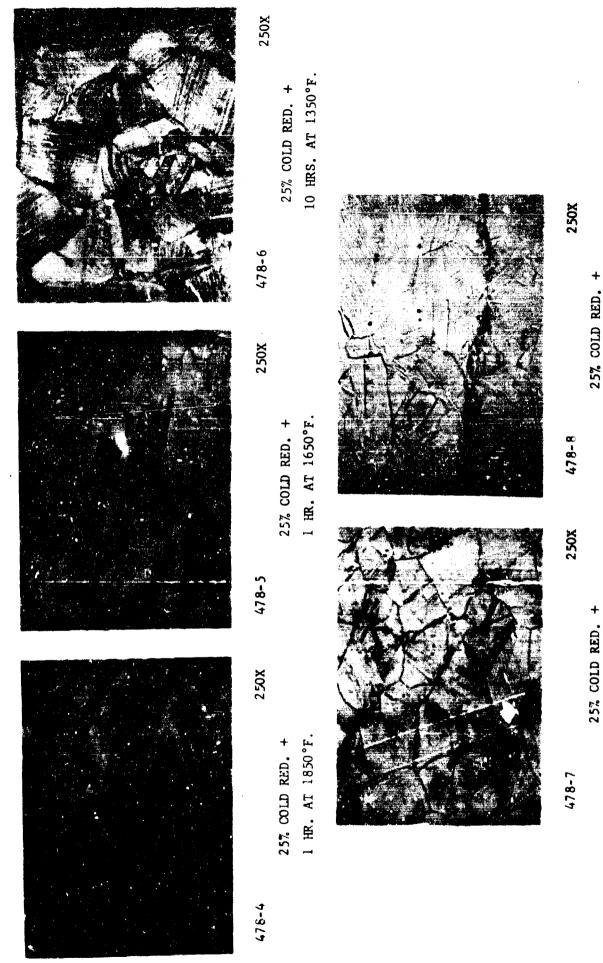


FIGURE 15. EFFECT OF COLD WORKING AND AGING ON MICROSTRUCTURE OF L-605.

10 HRS. AT 700°F.

1 HR. AT 1200°F.

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TABLE X

X-RAY DIFFRACTION DATA

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- Very small -
Very
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2150
25
2150
H15
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One hour at temperature, water quenched Air cooled
Dashes indicate no measurable precipitate.

Phuse Extraction Methods

The electrochemical extraction and x may diffraction analysis were performed by Sloan Research Industries, Santa Barbara, California using the -rehniques discussed by Wlodok $^{(2)}$. The general procedures set forth in this paper were followed, but it was found necessary to incorporate additional recipitate. Initial electrolytic extractreatments for turther retining of totions were performed using Beath Kit Model 1P 20 regulated power supply which could be adjusted for the proper current density of 200 ma it 4 volts in a 10% sulfuric acid/methanal solutio Approximately 20 hours were required to digest 3 grams of the base alloy. In all cases this gave less than 5 mg of precipitate residue. Some problem was encountered with nonadherent scaling of the spectrographic grade graphite enthodes, resulting in plated material dropping into the bath. X-ray diffraction s.k., ed these flakes and dendrites to be of an a-cobalt structure which were removed from the bath with a magnet. After a sufficient quantity of alloy was electrolytically digested, the mixture was centrifuged and washed with a fresh quantity of 10% sulturie acid/methanol. This effectively dissolved any remaining cobalt particles left This treatment was followed by additional centrifuging and washing with distilled water. The acid free precipitate was spread on clean microscope slides and allowed to dry prior to preparation for the diffraction analysis.

Portions of the residue were additionally treated with a 10% bromine/ methanol solution by seaching for 24 hours. This more active agent was to dissolve any intermetallies present and leave only the inert carbides as residual precipitates. After centrifuging and washing with distilled water, this residue was dried for diffraction studies.

b. X-ray Diffraction Sample Proparation

The initial diffraction studies were performed by placing the dried residues into microsample holders for study with a Novelec X-ray diffractometer.

However, because of the small quantity of available precipitate, the intensity of the diffraction—guals were insufficient for all but three samples to be sunty sed with the diffractometer. Therefore, powder camera techniques were used because of their greater suitability for interessamples. Samples for the camera analysis were prepared by mixing portions of the extracted residue with a five percent collulose nitrate simple sectate solution and rolling the partially direct mixture into a 0.2 mm diameter fiber. A proctation before Scherrer powder diffraction camera was used, however, due to the x-ray fluorescence of the cobalt, nickel and from in the sample which caused excessive fogging of the film, it was necessary to one the lower intensity cobalt is a primary radiation for some of the sludies.

Target	Copper	Cobalt
Filtor	Nickel	Iron
Kilovolta Potential	40	30
Militamps	20	15
Pinholes	, 025 -, 025 TS	88
Film Type	llford Industri	al G Type No Sereen
	X Ray Film .	
Exposure	8 26 hours	
Development	6 minutes 68°	F in Eastman X Ray
	Developer	

c. Analysis of Hot Working Studies

Four samples from heat number 5 representing various conditions of hot work, i.e., 25% reduction at 2150°F with three of the samples being subsequently reduced 25% at 2150°F, 2000°F and 1800°F, were evaluated by the extraction/x-ray diffraction technique to investigate the effect of forging temperature on the local tion of embrittling precipitates. Prior to forging the material had been annealed for one hour at 2150°F. Results of the diffraction studies are in agreement with the appearance of the microstructures (Figures 5, 6) and the mechanical properties (Tables III, IV) of the hot worked material. The lower temperature working (1850°F)

and 9000°F) resulted in the formation of the $M_{\tilde{b}}$ C carbides with a tince amount of thems than 3% of the total earlinds) $M_{\tilde{c}\tilde{b}}$ C formed in the 1950°F forged sample only. The sample reforged at 2450°F contained a very small amount of $M_{\tilde{b}}$ C indicating that these temperature is sufficient to retain most of the earlides in solution during a second reheat. No measurable amount of any precipitate was found in the sample with the single 25% reduction at 2450°F indicating that the precipitate formation occurred during the second forging operation. The ductifity of the material as measured by clongation and reduction of area. (Table IV) show a decrease from 46 o and 40.9, respectively, for the single reduced material to 34.0 and 32.6, respectively. For the samples reforged at 1850°F. The hardness of the samples (Table IV) also increased from $R_{\tilde{C}}$ 31 to $R_{\tilde{C}}$ 41. Although a major portion of the change in mechanical properties can be attributed to the effect of relative degrees of working of the material, the presence of the carbides should as a boseible contributing factor.

d. Analysis of Cold Working Studies

Four 25% cold worked samples from heat number a were evaluated by the extraction x-ray diffraction technique to investigate the effect of amealing temperature on the formation of precipitates. The material was annealed at 2150°F for one hour and water quenched prior to the cold reduction. One sample was evaluated in the as-worked condition and the remaining three were evaluated after annealing at 2250°F, 2150°F and 2000°F for one hour followed by a water quench. The microstructures of the as-worked sample and the two with the higher temperature anneals. (Figure 8) are relatively free of visible intergranular or grain boundary precipitates. The extraction x-ray diffraction analysis confirmed this observation showing no measurable amount of precipitate phase present. The microstructure of the sample annealed at 2000°F has a heavy grain boundary recipitate as well as several large into granular carbides. The extraction x-ray diffraction analysis showed the presence of considerable amounts

of M₆C in this sample. The 2150°F treatment appears optimum from the standpoint of preventing precipitate formation without excessive grain growth as discussed in Section II)-3.

e. Analysis of Aging Studies

The formation of a Laves phase, probably (Co, Ni), (Cr, W) and commonly referred to as Co,W, has been shown by Wlodek (2) to increase with time during aging with a considerable decrease in ductility. Schulz (5) indicated that the number of lattice discontinuities present in the form of twins can contribute to the less of ductility by allowing the Laves or carbide precipitates to form in an adverse manner such as continuous platelets. The microstructure of the aged samples (Figure 15) show an increase in the density of slip planes or twins with increasing aging temperature and were assumed to be visible as a result of precipitate formation. Three samples which had been annealed at 2150°F for one hour and water quenched then cold rolled to a 25% reduction with subsequent aging for one hour at 700°F, ten hours at 700°F, and one hour at 1200°F were evaluated by the extraction/x-ray diffraction technique for the presence of embrittling precipitates. These samples were selected as representing the recommended aging treatment for the SCP/LASRM combustor center sections. No measurable amount of precipitate was found in either of the samples aged at 700°F or 1200°F for one hour. A measurable amount of Laves phase was found in the sample aged for ten hours at 700°F; however, the microstructure (Figure 15) does not indicate the presence of a precipitate phase. An electron microscopic examination was performed on this sample in an attempt to determine the extent and location of the Laves phase. The specimen was found to be composed of large grains showing considerable slip from the cold reduction. The grains were uniformly peppered with a very small (1000 - 2000 A°) Laves precipite. No intergranular precipitation was observable in the sample. Additional aging would be required to cause the particles to form the platelets which are commonly

described as the morphology of the Laves phase. Wlodek's work regarding formation of the Laves phase involved the evaluation of material aged at 1600°F for up to 1000 hours with a minimum exposure of 16 hours.

6. SPECIFICATION FOR FORGED PREFORMS

All the mechanical property data from the hot working and cold working studies were analyzed, particularly with respect to ductility, in an attempt to isolate an optimum composition for maximum workability. No one heat appeared outstanding, although significant trends were observed with respect to iron and silicon contents. High silicon is definitely undesirable for maximum fabricability. The effect of iron is not as pronounced although the somewhat scattered data indicate that iron has a beneficial effect. These results are in fair agreement with previous work performed by others (2-4). The effect of manganese was uncertain due to conflicting comparisons of data although it is believed that further work might prove that manganese is beneficial. The clemistry selected is shown in the following specification. Although the air meited heat (No. 6) was comparable with the others in many respects, difficulties experienced with this heat in hot forging and cold rolling (which was partially due to a difference in bar geometry compared to the others) precluded a decirton to select air melting as an optimum melting process. Based on these considerations, previous Marquardt experience, and additional data developed in Phase of this program, the specification included below was developed for forgings and forging stock of shear spinning quality.

TITLE: COBAL * ALLOY (L-605) FORGING STOCK AND FORGINGS OF SHE *R SEINNING QUALITY

1. Scope

The purpose of this specification is to establish material conditions, restrictive requirements and quality control procedures for L-605 cohalt base forging stock and forgings suitable for forming by shear spinning techniques.

2. Applicable Specifications

The following specifications and standards, of the issues in effect on date of invitation for bids, or as stated on the purchase order, form a part of this specification.

- 2.1 AMS 2261; Tolerances Nickel, Nickel Base and Cobalt Base Alloy Bars and Forging Stock.
 - 2.2 AMS 2630; Ultrasonic Inspection
- 2.3 ASTM E 45; Recommended Practice for Determining the Inclusion Content of Steel.
- 2.4 ASTM E 112 Methods for Estimating the Average Grain Size of Metals
- 2.5 Federal Test Method Standard No. 151a Metals; Test Methods

3. Technical Requirements

3.1 Composition - The composition of the alloy shall conform to the following:

Element	Percent	Check An	alysis
		Under Min.	Over Max.
Carbon	0.10 max	****	0.01
Manganese	1.00-2.00	0.04	0.04
Silicon	0. 25 max		0.01
Iron	1. 60-3. 00	0.05	0.10
Phosphorus	0.01 max	-	0.005
Sulfar	♦. 115 max	-	v. vo5
Chrisnian	17. 66-21.00	0. 25	0. 25
Micke)	9, 29-11.00	0. 15	0. 15
Camps tor	14.00-16.00	0.10	0.10
Cobalt	Remainder	-	

3.2 <u>Meiting Priction</u> - The material shall be produced by multiple vacuum melting process. So The final process shall be the vacuum consummable electrode remelting technique.

- 3.3 <u>Forging Practice</u> Forging parameters shall be chosen such that following solution hear treatment the forgings shall meet the requirements of Sections 3.6, 3.7, 3.8, 3.9 and 3.10. Ingots shall be reduced sufficiently in cross section to assure proper uniform refinement of structure in the forged billet.
- 3.4 Heat Treatment The forgings shall be solution treated by heating between 2150°F and 2200°F, holding at temperature for not less than 1 hour nor more than 3 hours per inch of cross section and quenching in agitated water. These parameters may be varied as required to meet requirements of Sections 3.6, 3.7, 3.8, and 3.10.
- 3.5 <u>Condition</u> Forgings shall be supplied in the solution annealed and descaled condition.
- 3.6 <u>Hardness</u> Forgings shall have a hardness not higher than Brinell 248 (Rockwell C 24).
- 3.7 <u>Tensile Properties</u> Tensile properties of forging stock and forgings in the solution annualed condition shall be:

Tensile Strength, psi 125,000 min.
Yield Strength, 0.2% offset 45,000 min.
Elongation, % in 4D 30 min.

- 3.8 <u>Grain Size</u> The grain size of forgings as determined by ASTM E-112 shall be predominantly 3 or finer with occasional grains as large as 2 permissible.
- 3.9 <u>Macrostructure</u> Flow line patterns of forged parts shall be as specified on the forging drawing.
- 3.10 <u>Microcleanliness</u> The procedure for determining the inclusion rating shall be in accordance with ASTM E-45, Method D. This rating based on specimens representing the worst area of inclusions shall not exceed the following:

Inclusion Rating

Type Inclusion A B C D

Thin 1.5 1.5 1 2

Thick 1 1 1 1.5

In addition, the material shall be substantially free of grain boundary precipitates when examined at 500X magnification after electrolytically etching with a solution of the following proportions: 100 ml $\rm H_20$, 40 ml acetic acid, 40 ml HC1, 15 ml $\rm H_2SO_4$, 40 ml HNO₃, and 25 g. FeCl₃.

- 3. 11 <u>Ultrasonic Inspection</u> The method of ultrasonic inspection shall be the immersion process in accordance with AMS-2630. All forgings (100%) shall be inspected to discontinuity indication limits of 3. 11. 1, 3. 11. 2, 3. 11. 3, and 3. 11. 4.
- 3.11.1 No discontinuity indications in excess of the response from a 3/64 inch diameter, flat-bottomed hole at the estimated discontinuity depth.
- 3.11.2 Multiple indications in excess of the response from a 1/32 in. diameter flat-bottomed hole at the estimated discontinuity depth shall not have their indicated centers closer than 1 inch.
- 3.11.3 Indications from a single discontinuity equal to or greater than the response from a 1/32 in. diameter flat-bottomed hole at the estimated discontinuity depth shall not be more than 1 in. in length.
- 3.11.4 Multiple indications shall not be of such size or frequency as to reduce the back reflection pattern to 50 percent or less of the back reflection pattern of normal material of the same geometry, with the crystal parallel to the front and back surfaces to insure that the loss of back reflection is not caused by surface roughness or part geometry variation.

- 3.12 <u>Workmanship</u> The material shall be uniform in quality and condition, free from pipe, flakes or heat cracks. It shall be free of defects such as seams, laps, cracks, slag, hard spots, porosity, rolled in scale, fissures, gas cavities, and undue segregation which may be detrimental to the fabrication or performance of parts.
- 4. <u>Identification</u> Forgings shall be identified as indicated on the forging drawing or purchase order.

5. Quality Assurance

- 5.1 Lot A forging lot is defined as all forgings produced in one run from the same heat of material and heat treated in the same furnace load.
- 5.2 <u>Chemical Analysis</u> A sample from each heat of material represented in the shipment shall be analyzed to determine conformance to the chemical composition requirements of Section 3.1.

5.3 Tensile Properties

- 5.3.1 Two each room temperature, tensile tests shall be made in accordance with FTMS 151 on each forging lot represented in the shipment.
- 5.3.2 The test specimens shall be generated from forged coupons, (forged from the same heat of material as the represented forgings, given the same reduction and heat treated with the forgings from prolongations on production forgings, or a sectioned forging as designated on the forging drawing.
- 5.4 <u>Micro-Examination</u> One forging or forged sample from each forging lot shall be prepared and examined to determine grain size and microcleanliness.
 - 5.5 Hardness Each forging shall be checked for hardness.

6. Reports

The vendor shall furnish with each shipment three copies of a

certified test report of the results of tests for chemical composition, tensile, hardness, and grain size. This report shall include the actual hot working temperature and the final solution heat treatment of the forged parts. This report shall include the purchase order, material specification number, heat number, size, length and quantity from each heat.

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SECTION IV

PHASE II - MANUFACTURING PROCESS DEVELOPMENT

The objective of this phase was to develop a process for producing SCP/LASRM combustion chamber center sections, utilizing the technology generated in Phase I. This effort was subdivided into two steps. First, five small test cylinders were shear spun from forged preforms procured according to the recommendations resulting from the Phase I work. The intent of this effort was to verify the specified preform quality requirements by producing components requiring a large reduction (50%) during shear spinning. After this was accomplished, five SCP/LASRM components were produced from larged preforms of the same quality level. The process thus developed served as a basis for a shear spinning process specification.

In addition, for purposes of exploring a much cheaper process, one centrifugally east SCP/LASRM preform was procured and shear spinning was attempted.

1. SUBSIZE TEST CYLINDER FABRICATION

As mentioned above, the mitial step in Phase II was to verify that preform quality specified in Phase I was adequate for cold shear spinning. In the interest of economy, a small conical shaped part (Figure 16) was selected for this purpose because of existing available tooling for torging and shear spinning. In addition, this particular part required a 50 percent single reduction during shear spinning, which was greater than was planned for shear spinning the SCP/LASRM parts, (i.e. approximately 30% max, reduction per pass). Therefore, fabrication of this part was a realistic and severe test of the material's ability to be cold shear spun.

L-605 forging stock (2-3/4 in, dia, bar) was supplied to the forging vendor, Arcturus Manurecturing Corp., Oxnard, California, by their supplier,

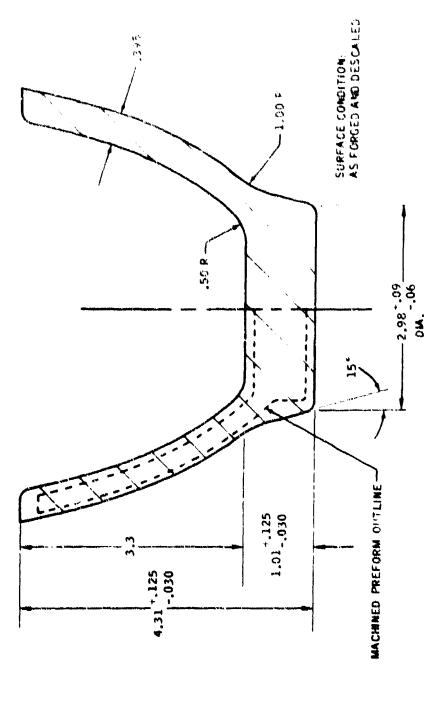


FIGURE 16. TEST CYLINDER PREFORM PORGING

Allyte Metals Company, Monroe, North Carolina. The double vacuum melted material was purchased to a specification equivalent to that included in Section III-6.

Examination of the hot rolled microstructure of the bar prior to forging revealed that it was heavily banded with earbide precipitates and contained a significant amount of grain boundary carbides. Annealing studies on samples of the material showed that a 2250°F, two hour annealing treatment followed by a water quench was effective in eliminating the grain boundary carbides and minimizing the carbide stringers. A decision was made to anneal the forging stock accordingly prior to forging.

The bar was then sectioned into 6-3/4 inch lengths as starting stock to produce the cup shaped forging shown in Figure 16. The forging operation consisted of the following sequence:

- (1) Preheat 2-3/4 in. diameter x 6-5/4 in. long bar to $2125 \pm 25^{\circ}$ F and hold at temperature for 15 minutes.
- (2) Upset using flat dies on a 12,000 lb. hammer to approximately 4-1/2 in, diameter by 2-3/4 in, long.
- (3) Reheat to 2125 ± 25°F and hold at temperature for 15 minutes.
- (4) Forge to final configuration using closed dies on a 10,000 lb.
- (5) Water quench.
- (6) Anneal at 2150°F + 25'F for 2 hours, water quench.

The five forgings were produced with no difficulty and shipped to Marquardt.

Representative samples were examined for grain size, microcleanliness, and hardness. The grain size determined by ASTM E-112 ranged from No. 3.5-4.5 which complied with the specification limit of No. 3 or finer with

occasional No. 2's permissible. The microcleanliness was determined per ASTM E-45, Method D, and was found to be acceptable based on the following data:

Inclusion Rating

Type	•	<u>A</u>	-	<u>B</u>	-	C	1	<u>D</u>
	Specified Max.	Actual	Specified Max.	Actual	Specified Max.	Actual	Specified Max.	Actual
Thin	1.5	1.3	1.5	0	1	0	2	0.6
Thick	1	0	1	0	1	0	1.5	0.3

The hardness data ranged from Brinell 237-269, (Rockwell C22-28). This was slightly marginal compared to the specification limit of the Brinell 248 (R_C 24) max. However, the higher readings (248-269 PHN) occurred on the base of the forging and not on the wall which is the only portion that is shear spun. The general appearance of the microstructure showed heavier than usual carbide stringers, but no evidence of grain boundary carbides which were considered detrimental for shear spinning operations and were prohibited by the specification. A typical photomicrograph of a forging is shown in Figure 17. The vendor's certified chemical analysis is shown in Table XI along with the specified composition.

The five forgings were machined into shear spinning preforms as shown in the sketch, Figure 16. The parts were shear spun at the Marquardt-Van Nuys facility using a horizontal 20,000 lb. single roller floturn machine (Figure 18). A single pass was made utilizing approximately a 50% reduction (reducing the wall from 0.215 in to \sim 0.118 in. in thickness). The shear spun parts are shown in Figure 19. The parts spun very well but with one minor problem. One of the parts showed significant cracking on the inner surface at the radius between the wall and base (closed end) of the preform, and two others showed a very superficial crack at exactly the same location. A cross section of the



548-3 250X

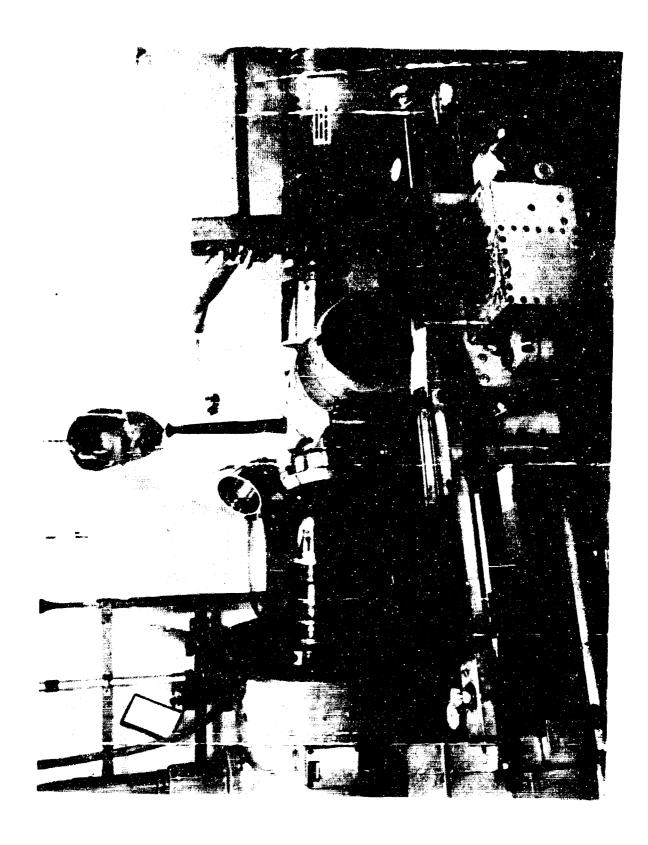
FIGURE 17. MICROSTRUCTURE OF ANNEALED SUBSIZE
TEST CYLINDER PREFORM
(ETCHANT: SCHANTZ)

COMPOSITION OF L-605 FORGING STOCK
(WEIGHT PERCENT)

Element	Requested	Vendor Analysis
C	0.10 Max.	0.095*
Si	0.25 Max.	0.23**
Fe	1.0-3.0	1.17
P	0.010 Max.	0.006
s	0.015 Max.	0.015
Mn	1.00-2.00	1.86
Cr	19.00-21.00	19.97
Ni	9.60-11.00	9.90
w	14.00-16.00	14.97
Co	Remainder	Bal.
<u> </u>		

^{*} Verified at Marquardt: 0.10, 0.095.

^{**} Verified at Marquardt: 0.2, 0.2.



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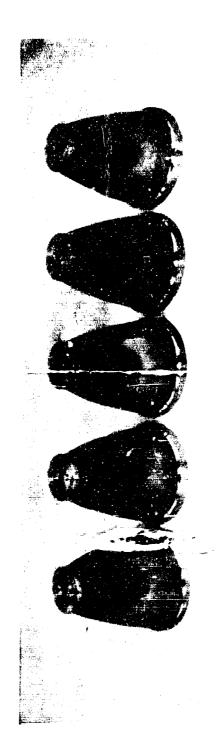


FIGURE 19. SHEAR SPUN TEST CYLINDERS

PRECEDING PAGE BLANN most severe crack is shown in Figure 20. The floturn mandrel was slightly worn in that location, which in combination with stack tolerances on the machined preforms could have caused the problem. The entire wall on each preform spun extremely well with no evidence of grain boundary separation or other surface discontinuity which indicated that the preform was of acceptable shear spinning quality.

Tensile specimens were machined from the longitudinal direction of one of the cylinders to investigate the effect of cold shear spinning on tensile properties and to determine uniformity of properties throughout the part. In addition, some of the samples were given the most promising aging treatment (as discussed in Section III-4, i.e. 700°. F for 10 hours) prior to testing. The data, listed in Table XII, showed very high strength and low ductility which demonstrates the extremely high work hardenability of L-605. The aging treatment was effective in increasing the yield strengths approximately 12-1/2%, but also reduced the low ductility to zero. As expected, the properties were very uniform compared to the data generated on cold rolled bar stock in Phase I of the program.

2. SCP/LASRM COMBUSTION CHAMBER CENTER SECTION FABRICATION

a. Preform Fabrication

Forging stock in the form of 8 in. diameter round-cornered square bar was supplied to Arcturus Manufacturing Corp. by their supplier, Allvac Metals Company from the same heat as the 2-3/4 in. diameter stock used for the subsize test cylinders.

Examination of the microstructure at Arcturus prior to forging revealed an appearance similar to that of the 2-3/4 in. diameter bar, i.e., heavy banding and grain boundary precipitates. Also, a duplex grain size was evident near the periphery of the bar. Annealing studies were conducted concurrently with material from the 2-3/4 in. diameter bar as discussed in





Section IV-1. The same annealing treatment was selected for the 8 in. bar, i.e., 2 hours at 2250°F followed by a water quench.

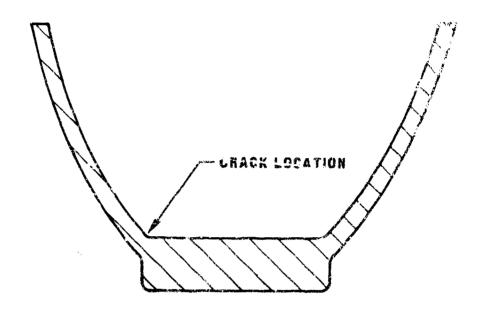
A sketch of the forging is shown in Figure 21 which also shows the outline of the machined shear spinning preform. Five of these parts were made as detailed in the flow chart, Figure 22.

Samples from the forgings were examined at Marquardt-Van Nuys to determine conformance to the procurement specification. It was found that the hardness was slightly above the specified limit (262-269 BHN vs. 248 max.)

The grain size was acceptable, ASTM No. 5 - 6.5, and the microclean-liness was the same as that of the small forgings as reported in Section IV-1.

An additional anneal had been planned after machining the preform prior to spinning. Based on the results from the six heats evaluated in Phase I and on other heats processed at Marquardt, an additional annealing treatment of 2150°F for 1 hour followed by a water quench had been preselected. However, since this particular heat did not respond typically to the first annealing treatment of 2150°F for 2 hours as evidenced by the grain boundary precipitates and higher than expected hardness, additional heat treating studies were conducted to optimize the post-machining anneal. Small pieces from the base of three forgings were annealed for 2 hours at 2150, 2175, and 2200°F and for 4 hours at 2175°F. The results indicated that the 2200°F, 2 hour annealing treatment would improve the microstructure and lower the hardness to a satisfactory level. Figures 23 and 24 show respectively the microstructure of an as-received forging and after annealing for two hours at 2200°F (followed by a water quench). Based on this information, these parameters were selected for the post-machining annealing treatment.

The forgings were machined to the sketch shown in Figure 25. In process and final dimensional inspection was performed with the assistance of templates





548-3

50X

FIGURE 20. I.D. RADIUS CRACK IN SHEAR SPUN TEST CYLINDER

TABLE XII
TENSILE PROPERTIES OF SHEAR SPUN TEST CYLINDERS*

Spectmen No.	History	Yteld Strength (KSD	Ultimate Strength (I.SI)	Elongation (%)
L-2	Shear spun	237.8	271.2	2
L-4	Shear spun	233,4	267, 1	2
L-6	Shear apun	248,2	272.5	2
L-8	Shear spun	233, 9	270.1	2
L-10	Shear spun	243.4	271,9	2
	Average;	239,3	270,6	2
L-1	Shear spun aged **	264.5	290,9	0
L-3	Shear spun sgcd**	267.5	294,6	0
L-5	Shear spun aged **	285.0	289,5	0
L-7	Shear spun aged **	262.2	286.6	0
	Average:	269.8	290.3	0

^{*} Cold shear spun, 50% reduction

^{**} Aged at 700° for 10 hours.

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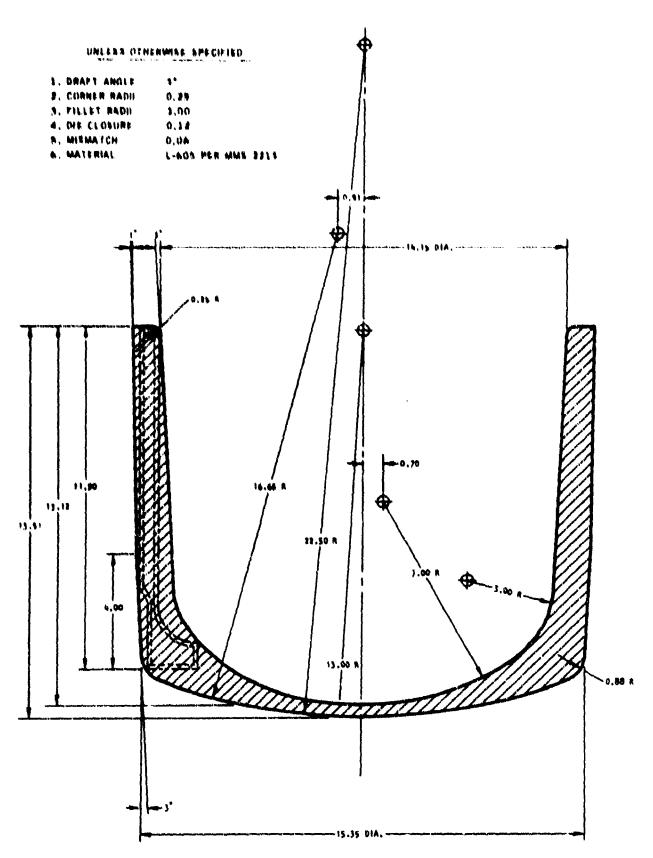


FIGURE 21. CENTER SECTION PREFORM FORGING

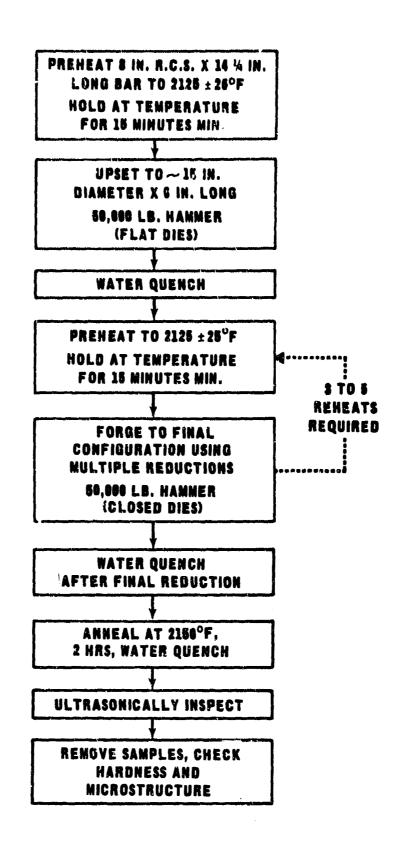
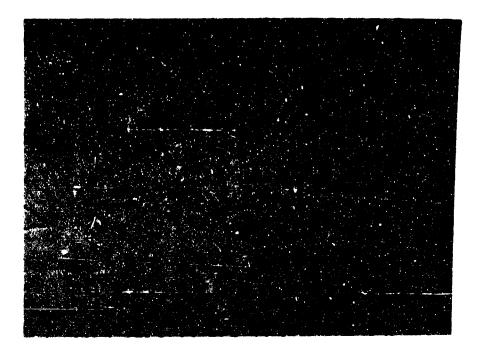
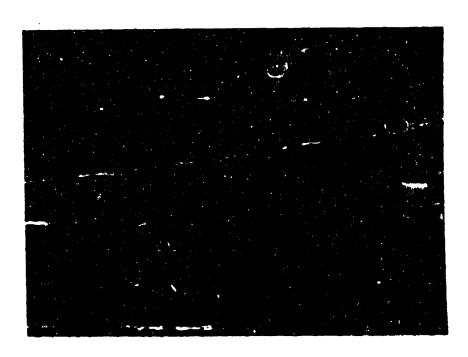


FIGURE 22. FORGING PROCESS FLOW DIAGRAM



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FIGURE 23. MICROSTRUCTURE OF AS-RECEIVED SCP/LASRM PREFORM (FORGED AND ANNEALED AT 2150°F, 2 HOURS, W.Q.) (ETCHANT: SCHANTZ)



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FIGURE 24. MICROSTRUCTURE OF ANNEALED SCP/LASRM PREFORM (SAME AS FIGURE 23 + 2200°F, 2 HOURS, W.Q.) (ETCHANT: SCHANTZ)

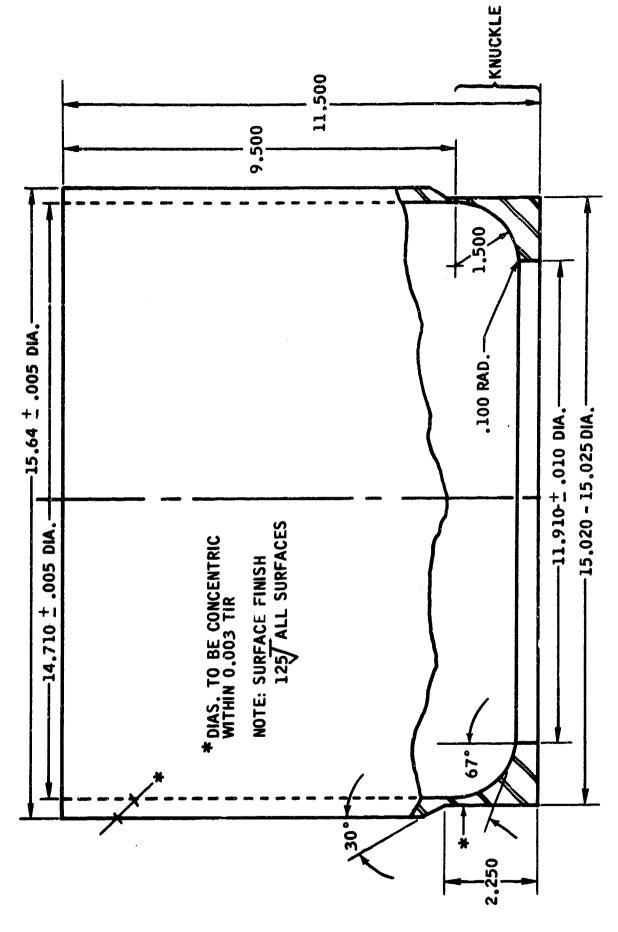


FIGURE 25. CENTER SECTION MACHINED PREFORM

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fabricated for this part. All preforms were successfully machined to the tolerances of the drawing.

The machined preforms were alkaline cleaned and individually solution annealed in air in a three zone electrically heated pit furnace. The parts were lowered vertically into the furnace; the flanged (heavy) end rested on a cruciform shaped fixture which supported the part in four places. No additional fixturing was utilized. The parameters of 2200°F for 2 hours followed by a water quench (as discussed above) were used. Temperature control of \pm 15°F or better was achieved for each part. The water quench was achieved by removal of the part and fixture with a crane and immediately submerging in an adjacent quench tank. No significant dimensional change resulted from this operation. The parts were then grit blasted, rinsed with water, and shear spun as described in Section IV-2-c. A photograph of a preform ready for spinning is shown in Figure 26.

b. Centrifugally Cast Preform

As an added scope to the program, a decision was made to investigate the feasibility of shear spinning a centrifugally cast preform along with the five SCP/LASRM forgings. The advantage of this approach would be a very significant cost savings compared to the die forging.

A casting was purchased from Centrifugal Products, Inc., Long Beach, California, in rough machined form, 15-1/2 in. O.D. x 12 in. long x 3/4 in. wall. A photograph of the casting is shown in Figure 27. Due to the tight schedule, it was necessary to procure the casting on a best efforts basis. The vendor 's analyses are shown in Table XIII and were within requested limits except for silicon which was only slightly high (0.30 vs. 0.25 max.). As requested, the vendor supplied X-rays of the casting which showed no internal defects, although unfortunately, the X-rays were not of optimum quality.

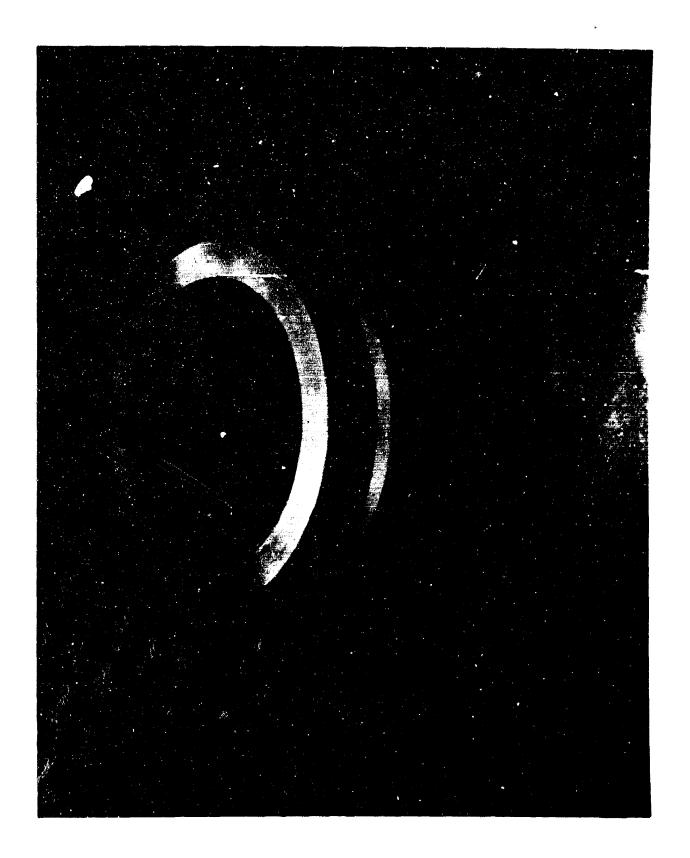


FIGURE 26. SCP/LASRM MACHINED, ANNEALED AND GRIT BLASTED PREFORM

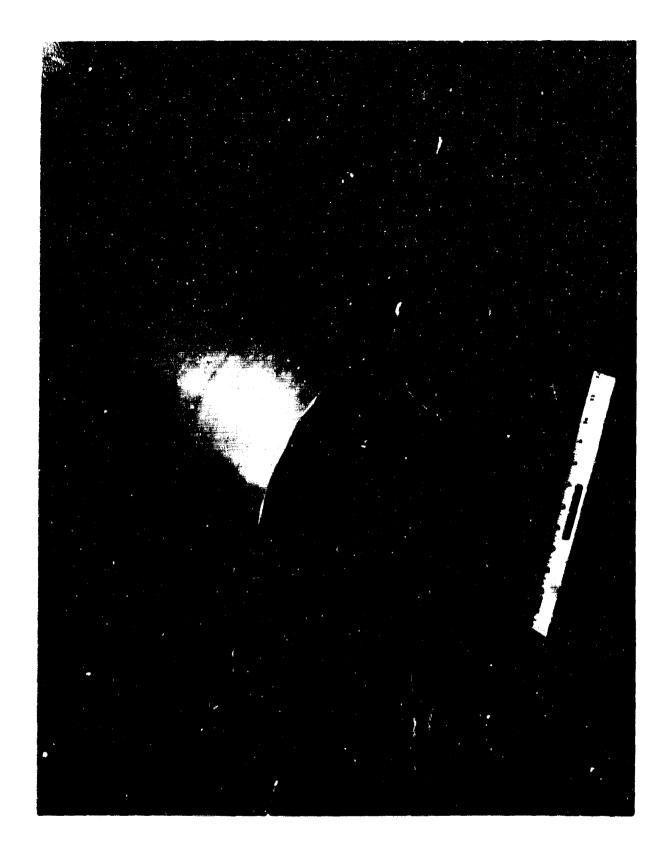


TABLE XIII

COMPOSITION OF CENTRIFUGAL CASTING

(WEIGHT PERCENT)

Element	Analysis
C	.10
Si	. 30
Fe	1.88
P	0.010
s	0.010
Mn	1.40
Cr	19.85
Ni	10.72
w	14.63
Co	Bal.



A section of the as-received casting was removed for hardness and metallography. The casting proved to be very soft (237 BHN). The microstructure, Figure 28, showed the typical centrifugal casting columnar grains extending from the outside diameter through about 15% of the wall. The higher magnification photomicrograph, Figure 28, showed segregation of low melting phases in the grain boundaries. Homogenization treatments of 2175 and 2200°F for two hours were not effective in significantly reducing this condition as shown in Figure 29. As a result, a decision was made to shear spin the casting without a homogenization treatment, particularly since the hardness was relatively low.

The casting was machined to the dimensions of the cylindrical portion of the preform sketch. Figure 25. An available knuckle forging of the same configuration as the knuckle portion of the forged preform, Figure 25, was TIG welded to one end of the finished machined cylinder. The preform was alkaline cleaned and an attempt was made to shear spin as discussed in Section IV-2-c.

c. Shear Spinning

As in all previous operations, the five parts were shear spun as a production lot rather than individually, since the primary objective was to demonstrate that the material could be consistently shear spun on a production basis, if proper process controls were exercised on every operation beginning with melting. The basic shear spinning parameters and tooling requirements were based on those utilized successfully on five out of 20 parts during the early SCP/LASRM program as discussed in Section I. Because that program had experienced difficulty in expanding-to-size after shear spinning, a decision was made to shear spin to size on this program and thus eliminate the expanding operation. Therefore, the preform and mandrel were made slightly larger, and essentially the same spinning parameters were used.

The parts were shear spun on a two-roller vertical Hufford Spin Forge Machine. This machine has a capacity of 225,000 lbs. on each roller.



I.D.

o.D.

561-2 5X

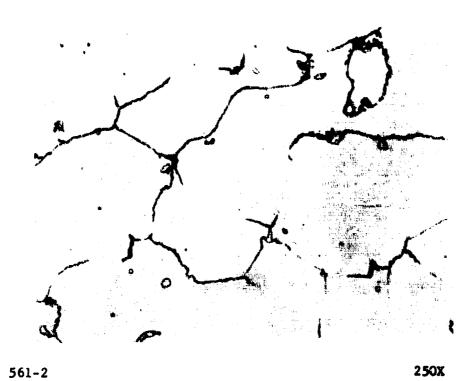


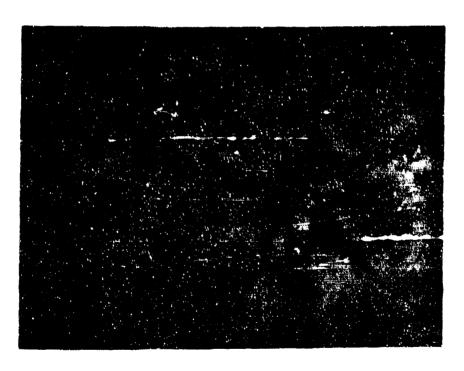
FIGURE 28. MICROSTRUCTURE OF L-605 CENTRIFUGAL CASTING. (ETCHANT: SCHANTZ)



561-2

250X

2175°F, 2 HRS., W.Q.



561-3

250X

2200°F, 2 HRS., W.Q.

FIGURE 29. MICROSTRUCTURE OF CENTRIFUGAL CASTING AFTER HEAT TREATING. (ETCHANT: SCHANTZ)

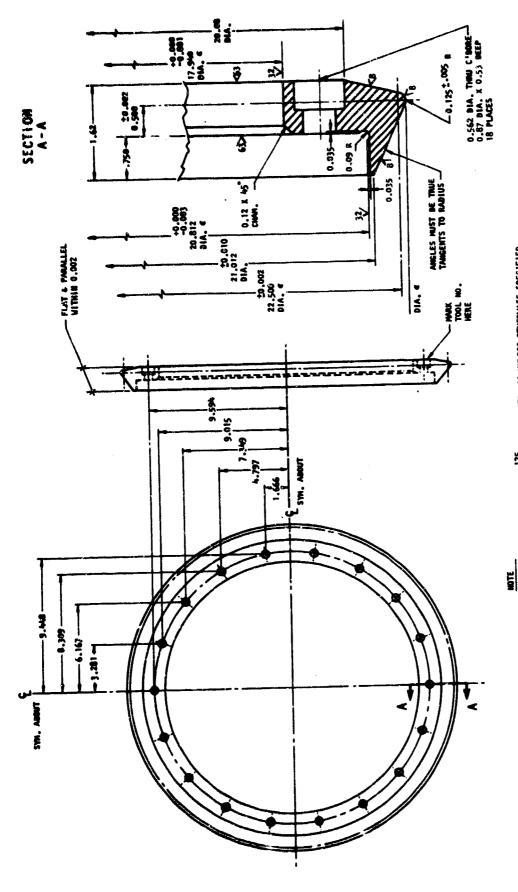
Sketches of the roller configuration and mandrel are shown in Figures 30 and 31 respectively. The angle of the rollers were set at 15°F with respect to the axis of the mandrel for all operations.

The basic shear form process utilized is cutlined in flow chart form, Figure 32. The 2185°F interstage annealing temperature was selected based on annealing studies performed on samples cut from one part after the shear spinning first pass. Microscopic examination showed that 2185°F for 1 hour followed by a water quench was effective in eliminating grain boundary precipitates without excessive grain growth. The hardness was reduced from 509 BHN on the asspun sample to 228 BHN by this treatment. The interstage annealing treatments were performed in the same furnace and by the same process described in Section IV-2-a. No significant dimensional change resulted from any of the interstage annealing treatments.

Details of the shear spinning parameters are shown in Table XIV.

All five parts were processed essentially the same through the first three passes. The fourth pass was varied for improved dimensional control as discussed below. All five parts shear spun very well with no evidence of surface cracking or other discontinuity detectable by dye penetrant inspection after each pass. An example of a shear spun part is shown in Figure 33. The success of the shear spinning operations points out the desirability of controlling the microstructure and hardness of the as-forged preform and also of the part during multistage shear spinning. The fact that this heat of material did not respond typically to thermal treatments previously established for other heats indicates that process specifications should include provision for controlling microstructure and hardness rather than specifying definite annealing times and temperatures.

Spot checks of the dimensions during the first three passes revealed a slight tendency for ring rolling, i.e., the part continued to grow away from the mandrel a small amount with each successive pass. After the third pass, the lower portion of each part was 0.040 to 0.060 in. greater in diameter than



1. ALL SUBFACES TO BE 125 PER HIL STD.-10 UNLESS OTHERWISE SPECIFIED.

2. DIA, COBED TRUS; """ TO BE CONCENTRIC TO EACH OTHER WITHIN 0.002 T.1.R.
3. DETAIL -101 STEEL TO BE VANABIUM ALLOY CO. POTOMAC "N" HOT FORM
TOOL STEEL, AFTER MACHINING HEAT TREAT TO R/C 48-52 POLISH & CODED
SURFACES.

FIGURE 30. SHEAR FORM ROLLER

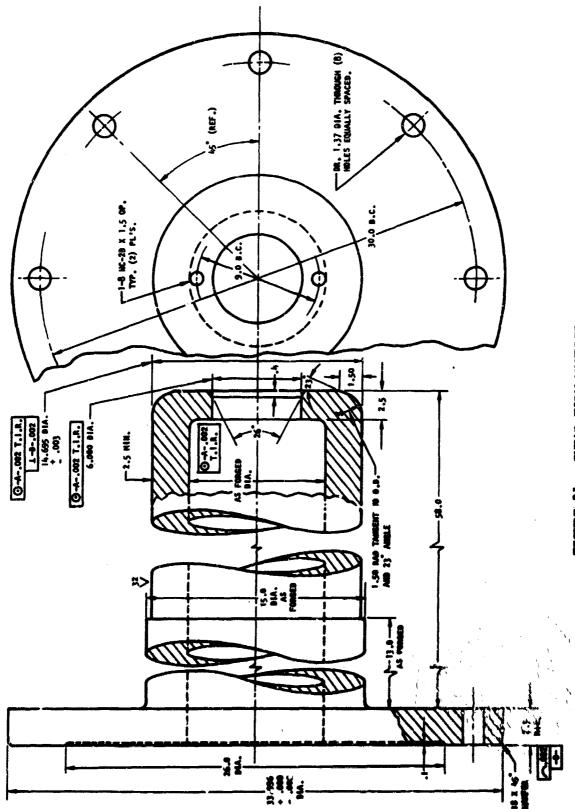
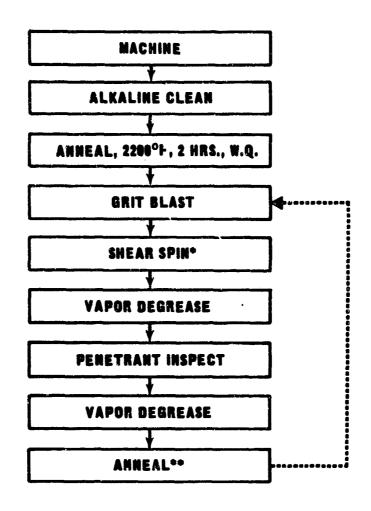


FIGURE 31. SHEAR FORM MANDREL



* SEE TABLE XIV FOR REDUCTIONS AND PARAMETERS

** BETWEEN 1ST AND 2 ND PASSES 2185 \pm 15° F, 1 HR., WATER QUENCH BETWEEN 2ND AND 3 RD PASSES 2185 \pm 15° F, 1 HR., WATER QUENCH BETWEEN 3RD AND 4 TH PASSES 2185 \pm 15° F, 45 MIN., WATER QUENCH

FIGURE 32. SHEAR SPINNING PROCESS FLOW DIAGRAM

SCPALASRM SHEAL SPINNING DATA TABLE XIV

				4	<u> </u>	8	2	3	¥	. H.	, i
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		*	Part Number	ج ا	_		\$		¥;	3 20 13 1	Į
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			4	8	•	Š.	2	8	Ą	•	ž
					J <u>€</u>	13	jįŝ	From From From From From From From From	128	112	

is amount of one roller lead over the other in inches. Her 3/6" radius, milling roller 1/4" radius.

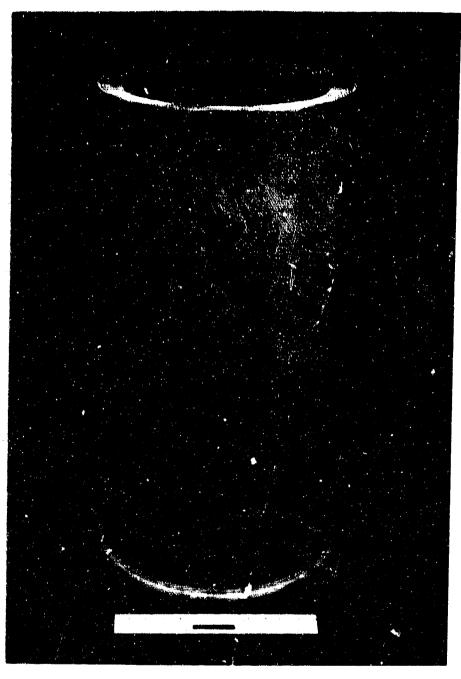




FIGURE 33. SCP/LASRM AS-SPUN COMBUSTION CHAMBER CENTER SECTION.

that of the mandrel. However, prior Marquardt experience had indicated that the fourth pass would remedy this situation, using the parameters listed for parts nos. 2 and 5, Table XIV, i.e., slower rotational speeds and faster feed rates. Upon inspection of these parts after the fourth pass, (Table XV) it was evident that these parameters were not completely effective in holding the bottom 6 in. portion of the cylinder within dimensions that would clean up to the tolerances of the drawing, Figure 34. However, an important factor should be noted here. A ring had been removed from each of these parts for in-process evaluation; otherwise the oversize portion (additional length) could have been cut off and discarded during actual machining of the part. In practice, the parts would have been useable, since the mating hardware exaltd have been machined to fit.

Further efforts were made to correct the oversize condition on parts nos. 1, 3, and 4 during the fourth pass using the following variations, detailed in Table XIV:

- (a) increased feed rate
- (b) even roller settings (no lead)
- (c) reduced rotational speed
- (d) smaller roller radii

Although these variations helped the situation, to varying degrees as can be seen by the data in Tubles XIV and XV, it was apparent that one or more of several obvious approaches should be considered for closer dimensional control in making this part in the future:

- (a) Start with a longer preform so that bell shaped oversize portion can be discarded.
- (b) Use a slightly smaller mandrel.
- (c) Reduce the total number of passes to two or three by increasing reductions thus minimizing ring rolling tendency, and also



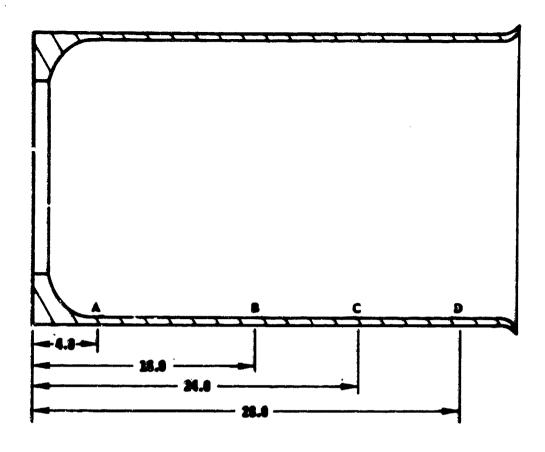


TABLE XV

DIMENSIONS OF SHEAR SPUN PARTS

Location	:	A	F	3	C		D	, 	Length (in)
Part No.	Wall (in)	O.D. (in)	Wall (in)	O.D. (in)	Wall (in)	O.D. (in)	Wall (in)	O.D. (in)	\ <u>\</u>
1	0.126	15.042	0.132	15.059	0.133	15.668	0.133	14.089	33-1/4
2	0.125	14.991	0.127	15.016	0.126	15.040	0.126	15.066	32*
3	0.131	15.027	0.131	15.097	0.132	15.148	0.124	15.168	29-1/8*
4	0.132	15.018	0.130	15.032	0.131	13.043	0.130	15.065	33
5	0.133	14.977	0.128	15.026	0.128	15.048	0.133	15.070	29 -7/84

^{*} Rings machined off bottom end for in-process evaluation.

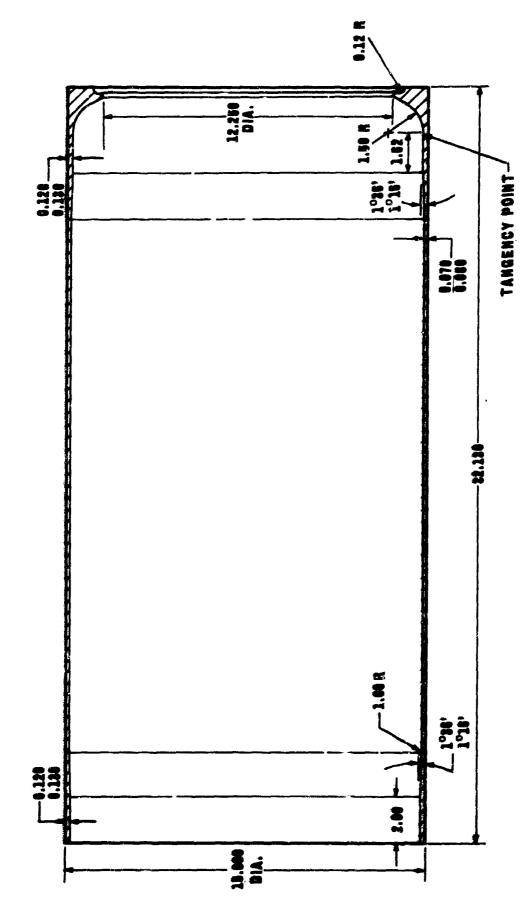


FIGURE 34. SCP/LASRM MACHINED CENTER SECTION

(d) Use relatively slow rotational speeds and high feed rates.

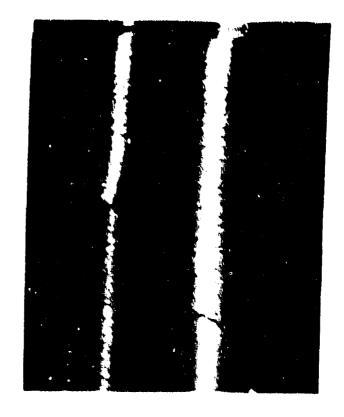
The centrifugally cast preform was given an initial reduction, using the same parameters as for Part No. 5, detailed in Table XIV. The wall thickness was reduced from 0.460 to 0.330 in., which resulted in approximately a 28% reduction. The results were gross cracking as shown in Figure 35.

Metallographic examination of cross sections taken through cracked areas, (Figure 36) confirmed that the cracks resulted from grain boundary separation as suspected.

Subsequent discussion with the vendor indicated they believe it is possible to improve the structure of centrifugal castings and minimize the grain boundary low melting phase by using improved equipment. Whether an improved microstructure would be adequate is unknown, since shear spinning is a very severe working operation and cannot tolerate any appreciable grain boundary weakness or discontinuity. No further effort was expended on the centrifugally cast part.

d. Evaluation of SCP/LASRM Parts

After the dimensional inspection described in the preceding section was completed, one of the parts (No. 2) was sectioned, and ten tensile specimens were removed from the mid portion of the chamber — five from the longitudinal and five from the circumferential direction. Half of the specimens were given the 700°F, 10-hour aging treatment discussed in Sections III-4 and IV-1. The results, listed in Table XVI showed that the transverse strengths were significantly higher than the longitudinal strengths, and that the transverse ductilities were correspondingly lower. The hoop stress design requirements of the part require 175 KSI room temperature yield strength, which was not met by the as spun parts. The aging treatment was very effective in raising the transverse properties to this level with a minimum effect on ductility. Based on this information, a decision was made to age the remaining cases prior to pressure proof testing.







0605-1 50X



0605-2 100x

FIGURE 36. PHOTOMICROGRAPHS SHOWING INTERGRANULAR CRACKING OF SHEAR SPUN CENTRIFUGAL CASTING

TABLE XVI

ROOM TEMPERATURE TENSILE PROPERTIES OF SHEAR SPUN SCP/LASRM COMBUSTION CHAMBER CENTER SECTION

(20% Cold Worked)

Specimen No.	History	Orientation	0. 2% Y. S. (KSI)	u. t. s. (KSI)	Elongation (% in/in)
827N	A aa min	Circumferential	163.4	210.3	7
	As -spun	Circumferential	145.2	210. 3	
828N	As-spun				8
829N	As-spun	Circumferential	156.5	212. 2	7
852N	Spun + 700°F-10 hr. age		187.0	230.8	6.5
853N	Spun + 700°F-10Hr. age	Circumierential	191.3	224. 9	6
830N	As-spun	Longitudinal	153. 1	206. 2	16
831N	As-spun	Longitudinal	156. 0	206.5	14
854N	Spun + 700°F-10 hr. age	Longitudinal	174.8	207. 7	13
855N	Spun + 700°F-10 hr. age	Longitudinal	171.7	209. 6	10
856N	Spun + 700°F-10 hr. age	Longitudinal	175.6	208. 2	10

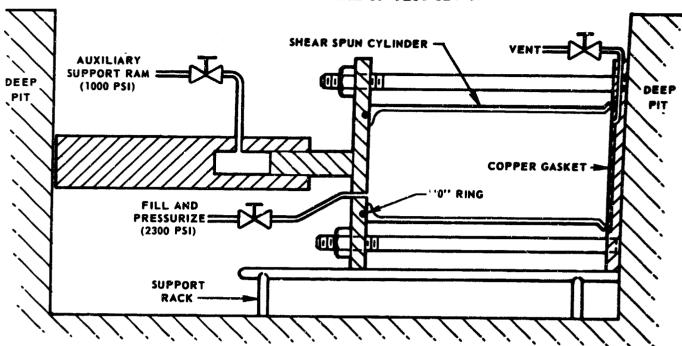


The remaining four parts (Nos. 1, 3, 4 and 5) were ultrasonically inspected at Ultrasonic Associates, South Gate California, at a sensitivity sufficient to detect any indication equal to or greater than a 3/64 in. diameter flat bottomed hole, per the specification requirements (Section IV-3). All four parts were 100% tree of any indications.

Four of the five shear spun cylinders were subjected to a proof pressure test equivalent to that specified for the SCP/LASRM compustion chamber. The SCP/LASRM hardware is pressure tested in the final configuration i.e. after welding the forward dome and expansion nozzle to the center section. For this program, only the center section was tested in the as-spun (not machined) condition. The test pressure was increased from 1375 psi (used for machined cases with a 0.070 - 0.080 in. wall) to 2300 psi to compensate for the thicker wall (0.125 inch) of the as shear spun cylinders. The specified 20 second hold time remained unchanged.

The tests were performed by Hydro-Test Corporation of Long Beach, California. The test fixture, supplied by Hydro-Test consisted of two blind flanges with a fill and vent system and eight threaded rods to seal the flanges against the cylinder as shown in Figure 37. The flange in contact with the flared end of the cylinder was sealed with a soft copper gasket and the other flange was sealed against the flat portion (knuckle) of the machined preform with an "O" ring. Approximately 2400 ft lbs. torque was required to seal the flanges. In order to avoid excessive loading and possible buckling of the non-supported cylinder in the test fixture, a hydraulic cylinder pressing on the forward flange was pressurized to 1000 psi after the cylinder was filled with water and leak checked at an internal pressure of 500 psi. The external load provided the additional force to maintain the seals at the final test pressure. The external pressure also compensated for the tensile stress applied to the bolts and reduced the possible loss of internal pressure because of elongation of the bolts. The internal pressure was increased to the required 2300 psi proof pressure and

SCHEMATIC DIAGRAM OF TEST SET-UP





TEST OF CYLINDER NO. 5

FIGURE 37. PRESSURE TEST SET-UP

held for 20 seconds. All four cases passed the test with no significant yielding as verified by a spot check of dimensions before and after testing.

3. SPECIFICATION FOR SHEAR SPINNING

Based on the information generated in shear spinning the five test cylinders and the five SCP/LASRM combustion chamber center sections during this program, and based on previous Marquardt experience, a process specification for shear spinning L-605 was prepared. This specification is of a general nature rather than for a specific part, and serves as a basic document for quality control of critical variables in L-605 shear spinning processes. Appendices can be added for specific parts, such as the SCP-LASRM combustion chamber center section. Such an appendix for this part will not be written at this time since further effort to refine the shear spinning parameters for dimensional control is necessary as discussed in Section IV-2-c.

TITLE: SHEAR SPINNING OF COBALT BASE ALLOY (L-605)

1. Scope

This specification establishes the quality control requirements for the cold shear spinning of L-605 hardware from forged preforms.

- 2. Applicable Specifications
 - 2.1 MIL-I-6866 Inspection, Penetrant Method
 - 2.2 AMS 2630 Ultrasonic Inspection
- 2.3 ASM E-112 Methods for Estimating Average Grain Size of Metals
- 2.4 Marquardt Specification "Cobalt Alloy (L-605) Forging Stock and Forgings of Shear Spinning Quality" *
- 3. Technical Requirements
- 3.1 <u>Material</u> The preform forgings shall conform to the requirements of Section 2.4.

^{*} Included in Section III - 6 of this report.

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- 3. 2 Machining
- 3. 2. 1 Surface Finish The preforms shall be machined to produce a surface finish of 125 rms or better.
- 3. 2. 2 Dye Penetrant Inspection Machined preforms shall be dye penetrant inspected in accordance with Section 4. 1.
- 3. 2. 3 <u>Hardness</u> The machined preforms shall be checked for hardness to determine effect of machining on surface hardness. If the surface hardness exceeds Rockwell C 26, preforms shall be annealed in accordance with Section 3. 4. 1 prior to shear forming.
 - 3.3 Shear Forming
- 3. 3. 1 Parameters The parameters for shear forming shall be established for each particular part. The established parameters shall be maintained in order to reproduce acceptable hardware of consistent quality.
 - 3, 3, 1 Interstage Inspection
- 3.8 %. Dimensional First article shall be dimensionally inspected for contentance to drawing requirements and verification of persent reduction. Additional measurements shall be taken during a production run to assure quality control.
- 3. 3. 1. 2 Penetrant All parts small be penetrant inspected in accordance with Section 4. 1 after each phear forming operation.
- 3. 3. 2 A terretage 6 to cal Parts shall be annealed in accordance with Caption 3. 4. 1 after each intermediate shear forming operation.
 - 3.3.3 Post Shear Spinning Inspection
- 3.3.3.1 <u>Dimensional</u> All parts shall be subjected to 100% dix a national inspection for conformance to the requirements of the engage drawing.
- 3. 7. 3. 2 <u>Fenetrant</u> All parts shall be penetrant inspected in accordance with Section 4.1.

- 3.4 Heat Treatment
- 3.4.1 Amosling
- 3. 4. 1. 1 When required, the parts shall be subjected to an annealing cycle that will assure:
 - (a) An ASTM grain size of 3 or finer with an occasional 2 permissible,
 - (b) maximum hardness of Rockwell C 26,
 - (c) a microstructure substantially free of grain boundary carbides.
- 3. 4. 1. 2 Parts shall be cleaned by vapor degreasing or equivalent prior to annealing.
- 3. 4. 1. 3 Annealing shall be performed in an air atmosphere furnace controlled to an accuracy of $\pm 15^{\circ}$ F of the required temperature necessary to meet the requirements of Section 3. 4. 1. 1.
- 3. 4. 1. 4 Parts shall be water quenched from the assealing temperature and grit blasted to remove any heat treat scale.
 - 3, 4, 2 Aging
- 3. 4. 2. 1 When required, the parts shall be subjected to an aging cycle such that the finished parts will meet the requirements of the engineering drawing.
- 3. 4. 2. 2 Parts shall be cleaned by vapor degressing or equivalent prior to aging.
- 3. 4. 2. 3 Aging shall be performed in an air atmosphere furnace controlled to an accuracy of \pm 25°F.
 - 3. 4. 2. 4 Parts shall be air cooled from the aging temperature.

4. Acceptance Requirements

- 4.1 Penetrant Inspection
- 4.1.1 All parts shall be subjected to 100% penetrant inspection in accordance with MIL-I-6866 as required by Sections 3.3.1.2 and 3.3.3.2.
- 4. 1. 2 Indications considered detrime o subsequent shear forming shall be cause for rejection at intersta, s inspection.

 Acceptance creteria on finished parts shall be as specified on the engineering drawing.
 - 4, 2 Ultrasonic Inspection
- 4. 2. 1 Ultrasonic inspection requirements and acceptance criterion shall be specified on the engineering drawing.
- 4. 2. 2 Ultrasonic inspection if required shall be performed in accordance with AMS 2636.
 - 4.3 Proof Pressure Testing

Proof Pressure testing requirements and acceptance criterion shall be specified on the engineering drawing.

4. 4 Destructive Testing

Tests to determine mechanical properties and general quality shall be performed as required by the engineering drawing.

SECTION V

CONCLUSIONS AND RECOMMENDATIONS

- 1. Shear spinning of L-605 forgings, (including SCP/LASRM chambers) can be successfully accomplished on a production basis if adequate quality control procedures are followed. Control of the microstructure and hardness is particularly important for shear spinning processes. The two specifications included in this report summarize the recommendations resulting from this program, i. e.
 - (1) "Cobalt Alloy (L-605) Forging Stock and Forgings of Shear Spinning Quality"
 - (2) "Shear Spinning of Cobalt Base Alloy (L-605)"
- 2. The variations in iron, silicon, and manganese contents of L-605 investigated in this program revealed no clearly defined effects of iron or manganese, but showed that silicon should be held to a maximum of $0.25 \, ^{\text{W}}/\text{o}$ for optimum hot and cold fabricability. Lowering the silicon to a very low level ($\sim 0.04\%$) revealed no added beneficial effects.
- 3. Limited data indicated that vacuum induction plus vacuum are remelted material is slightly more fabricable than air melted plus vacuum are remelted material. Further work is necessary to substantiate this.
- 4. The forging temperature for L-605 shear spinning preforms should be high enough to minimize grain boundary carbides and low enough for grain size control. The optimum temperature appears to be approximately 2150°F.
- 5. The optimum annealing time and temperature for L-605 varies from heat to heat of the same composition and with prior fabrication history. The annealing parameters for shear spinning applications should be selected to minimize hardness, grain growth, and grain boundary precipitates.

- 6. The room temperature strength properties of 20% cold worked L-605 can be increased significantly with a minimum decrease in ductility and and notch sensitivity by an aging treatment of 700°F for 10 hours.
- Shear spinning a centrifugally cast L-605 preform does not appear feasible,
 based on the single attempt made in this program.

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- G. D. Sandrock, R. L. Ashbrook, and J. C. Freche, "Effect of Variations in Silicon and Iron Content on Embrittlement of a Cobait Base Alloy (L=605)," NASA TN D=2989, September, 1965.
 September, 1965.
- 4. J. F. Nejedlik, "The Embrittlement Characteristic of a Low-Silicon Modified Cobelt-Base Alloy (L-605) at 1200 and 1600°F," TRW Elt 6870, A5 June 1866.
- 5. D. W. Schulz, "Post-Aging Ductility of Haynes Alloy No. 25," Presented AIME Symposium on High Temperature Alloys, Dallas, Texas, 27 February 1963.

APPROXIMATE HARDNESS CONVERSION NUMBERS

FROM ROJKWELL C TO BRINELL

Rockwell C Scale Hardness Number	Brinell Hardness Number 10 - mm ball 3000 kg load standard ball	Rockwell C Scale Hardness Number	Brinell Hardness Number 10 - mm bali 3000 kg load standard ball
58		37	344
54	-	36	336
83	•	35	327
52	800	34	319
51	487	33	311
60	475	32	301
49	484	31	294
48	451	30	286
47	449	29	279
46	432	28	271
45	491	27	264
44	409	26	258
43	400	25	253
42	390	24	247
41	381	23	843
40	371	22	297
39	362	21	231
38	353	20	226

Ref: G. L. Kehl, Principles of Metallographic Laboratory Practice, Metallurgy and Metallurgy Engineering Series, Third Edition p. 470, McGraw-Hill Book Co., Inc.

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This report summarizes a program designed to develop a process for producing large components for rocket/ramjet combustion chambers by cold shear spinning forged L-605 preforms. Phase I of the program involved work performed on six experimental heats of L-605 directed toward optimization of melting process, composition tolerances, fabricability, response to thermal treatments, and mechanical properties. This work resulted in an improved specification for L-605 forgings of shear spinning quality. Phase II of the program involved shear spinning process development for production of SCP/LASRM combustion chamber center sections which resulted in a L-605 shear spinning process specification. In addition, an attempt was made to shear spin a centrifugally cast preform.

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